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MEASUREMENT AND RANKING OF PERMEATION SPECIMEN THICKNESS PROFILES: HIGH-DENSITY POLYETHYLENE SWATCHES

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As part of the Explosive Destruction System for the Non-Stockpile Chemical Materiel Program, the Universal Munitions Storage Container was evaluated as a candidate for over-packing munitions (leaking and non-leaking) that contain chemical warfare agents for storage until destruction. A 1–2 year storage period was the program goal. Permeation experiments were performed with a series of material thicknesses over a 1–3 month period, and thickness versus breakthrough time was extrapolated to the 1–2 year target. Accurate measurement of permeation specimen thickness is important for the extrapolation of short-term permeation breakthrough times with thinner specimens to longer storage times for the munitions containers at full thickness. To meet this objective regarding thickness accuracy, about 3000 thickness measurements on 128 swatches were completed, statistically analyzed, and ranked. The most uniform specimens were selected for use in permeation testing. An exploratory study revealed that machining with an end mill produced adequate permeation specimens. The rather wide distribution of thickness values for any given set demonstrated that it was important to rank and select the specimens with the lowest variability for use in the test matrix. Overall, it is recommended that the swatches be accepted for use in the permeation experimentation.

15. SUBJECT TERMS

Permeation	Container	Thickness effect
Distilled mustard (HD)	Fick's law	Thickness uniformity
High-density polyethylene (HDPE)		

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PREFACE

The work described in this report was started in July 2013 and completed in May 2016. It was authorized under the Non-Stockpile Chemical Materiel Program, Explosive Destruction System, Universal Munitions Storage Container project.

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This report has been approved for public release.

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The author would like to acknowledge managerial support from Brian MacIver (U.S. Army Edgewood Chemical Biological Center [ECBC]; Aberdeen Proving Ground, MD) and James Horton, Lucille Forrest, and William Adams (U.S. Army Chemical Materials Activity; Aberdeen Proving Ground, MD). The author is grateful for useful discussions with W. Venner Saul (Sandia National Laboratories; Albuquerque, NM). Patricia Boone (Excet, Inc.; Springfield, VA) assisted in managing the permeation specimen inventory. The author is also thankful to Myat Win and Chad Gross (Engineering Directorate, ECBC) for the use of micrometers.

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CONTENTS

FIGURES

TABLES

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MEASUREMENT AND RANKING OF PERMEATION SPECIMEN THICKNESS PROFILES: HIGH-DENSITY POLYETHYLENE SWATCHES

1. INTRODUCTION

1.1 Objective

The Non-Stockpile Chemical Materiel Program is developing the Explosive Destruction System. The Universal Munitions Storage Container (UMSC) developed by Sandia National Laboratories (SNL; Albuquerque, NM) is a candidate for use in overpacking leaking and non-leaking munitions that contain chemical warfare agents (CWAs) for storage. Leaking munitions must be enclosed in storage containers made of materials of sufficient thickness that CWA permeation does not occur before a specified time, under a range of temperatures. A methodology for long-term permeation testing is required to support estimation of the maximum safe storage time for CWAs in containers of varying thicknesses. A storage time of 1–2 years is the program target. Permeation experiments must be performed with a series of thicknesses over a 1–3 month period to allow extrapolation of material thickness versus breakthrough time to the 1–2 year target. Container thickness is a critical factor in the permeation resistance of the containers. Accordingly, the accurate measurement of permeation specimen thickness is important for the extrapolation of short-term permeation breakthrough times to longer storage times.

To meet this objective for thickness accuracy, about 3000 thickness measurements on approximately 128 swatches were completed, statistically analyzed, and ranked. The most uniform specimens were selected for use in permeation testing. The results of the permeation studies are reported separately (*1*). In a parallel test program, the sorption of distilled mustard (HD) into high-density polyethylene (HDPE) was measured (*2*). The relevant test methods are those for permeation experimentation with hazardous chemicals (*3, 4*).

1.2 Review of Thickness Profiling Instrumentation

The fallback procedure for measuring accurate thickness values was to use a conventional micrometer and perform the measurements at predetermined locations on the swatch surfaces. In the interim, a survey was performed on an alternative method that was automated and could provide continuous three-dimensional thickness measurements, as a function of position, over the swatch surface. An example is given in Figure 1, which is a plot of material thickness versus position on the roll of protective material. The thickness over the 40 in. wide roll varied by about 10% (1.33/13.2 mil). This provided a measure of potential between-swatch variability. Over any 2 in. permeation swatch diameter, the thickness varied by several percent, providing a measure of within-swatch thickness variability. Access to automated thickness profiling instrumentation could not be obtained in time for this study; therefore, the manual method of thickness profile measurement was used. Instrumentation designs from various technology companies provide different measurement approaches to automated thickness profiling, such as the Filmetrics (San Diego, CA) F3-XXT system; SolveTech, Inc. (Wilmington, DE) precision gauges; Metralight, Inc. (San Mateo, CA) measurement products; and Elcometer (Manchester, U.K.) thickness gauges.

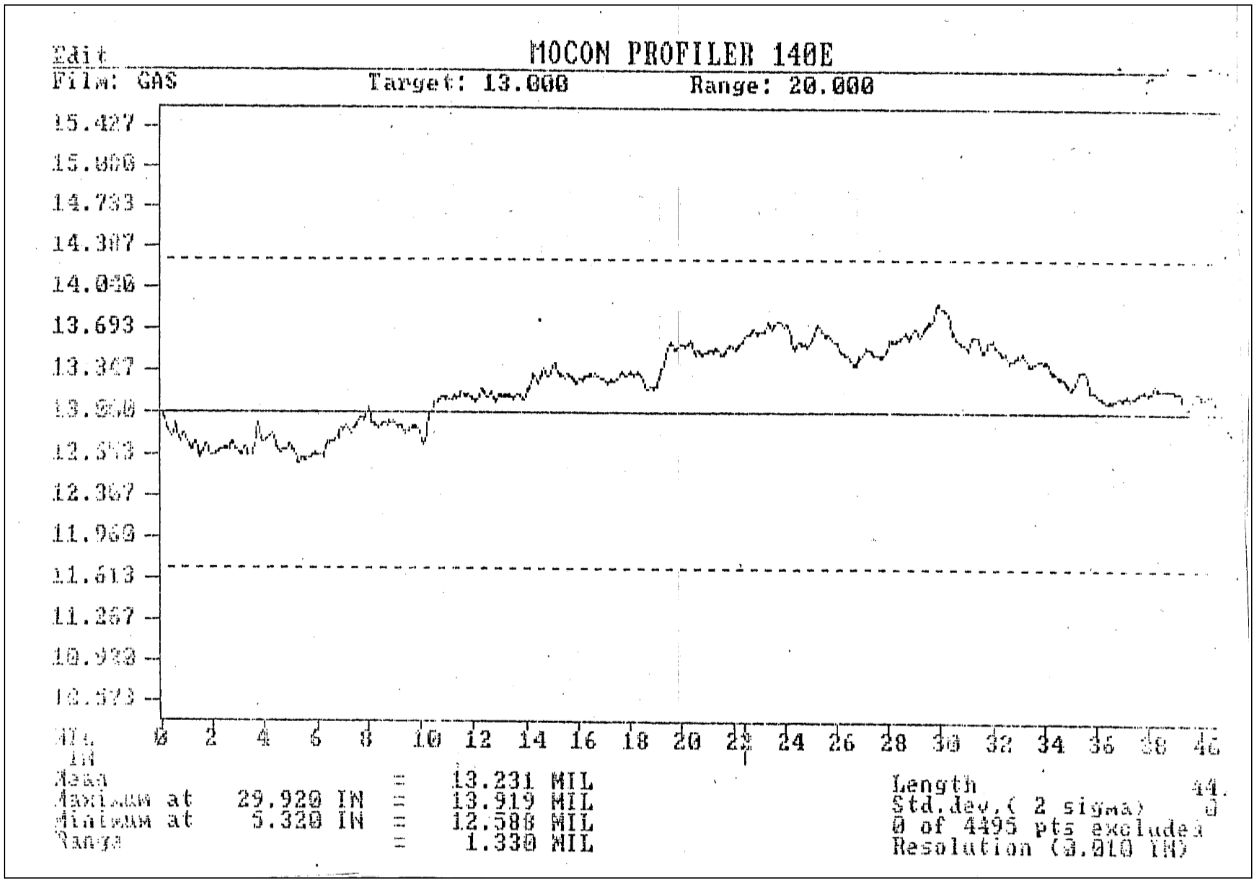

Figure 1. Example of automated thickness measurement as a function of position on protective material.

2. EXPERIMENTATION

2.1 Materials

The container material was composed of Dow Continuum DGDC-2480 BK bimodal polyethylene resin (Dow Chemical Company; Midland, MI). The UMSCs were produced from preformed pipe, and the end caps were machined from blocks of the same resin. The resin was composed of a >99% ethylene/hexene-1 copolymer with CAS no. 25213-02-9 polyethylene. All of the welded and non-welded swatches were machined at SNL and Innovative Plastic Solutions (IPS; Abingdon, MD), respectively.

2.2 Permeation Specimen Processing Options

Several methods were surveyed for producing permeation specimens of uniform thickness from either HDPE block or the actual HDPE pipe used in the munitions container. The most favorable options are listed in Table 1. Based on discussions with SNL personnel, the machining option was selected. Development of specific procedures for the machining process is described in Section 2.3.

Table 1. Options for Production of Permeation Swatches

Characteristic	Machine a "Well" for Liquid	Saw with Vacuum Clamping	Pipe Surface Sawing
Thinnest possibility: low; worst case	8 mil	40 mil; 40 mil	10 mil; 15 mil
Estimate (±)	5%	10%	10%
Increments feasible	5+	3	2
Advantages	Easier to clamp	Any diameter could be die-cut from the sheet	Inner pipe surface would be close to actual pipe surface; if outside is turned, remaining inside skin and surface are realistic
Disadvantages	Time, expense	Saw grooves; vacuum might be inadequate to retain specimen	If inside is turned, turned surface has no formed skin
Risk of not being able to deliver swatches with uniform thickness	Low	Low	Low

2.3 Development of a Machining Process for Permeation Specimens to Produce Uniform Thickness within a Swatch

The specimen production objective was to convert a block of HDPE into highly uniform permeation specimens that would fit into a permeation cell without leaking. Two trial swatches were produced (at IPS) by machining a cylinder-shaped well into a block of the Dow Continuum DGDC-2480 BK HDPE. A comparison was made between two milling techniques; one employed a coolant lubricant, and the other did not. These are referred to as "lube cooled" and "no lube", respectively. The use of a coolant lubricant is the standard technique for milling heat-sensitive thermoplastics. The no-lube technique was requested as an alternative to avoid sorption of nonpolar chemicals by the nonpolar HDPE, which can pre-swell a polymer and increase permeation of the test chemical.

Each HDPE block was first cut into slabs about 4 × 7 × 0.4 in. thick using a DoAll C-4A horizontal band saw (DoAll Company; Wheeling, IL). Each slab yielded three finished swatches.

The bottom surface of each slab was finished using a fly cutter on a Haas VF-2 CNC vertical mill (Haas Automation; Oxnard, CA). The fly cutter consisted of a large arbor from which extended a single-point cutting tool shaped like a lathe tool bit. This cutting tool extended downward and outward at an angle from the arbor. While the mill and arbor were rotated at slow to moderate speed, the slab was fed into the cutting tool, and the sharp edge of the cutting tool (jutting out from the arbor) contacted the swatch surface and removed HDPE with a wide circular motion.

After fly cutting was complete, five 1/4 in. bolt holes were drilled in each slab. These holes were located to apply equal clamp pressure to each disk without encroaching on the disk area.

The rectangular slabs were then clamped, bottom-surface down, to a fixture plate in a Haas VF-2 CNC vertical mill with size 1/4–20 bolts.

Using a 1/4 in. end mill and CNC programming, the slab thickness, the well, and the disk diameter were machined to the required dimensions. (Note that end mills are tools with cutting teeth at one end and on the sides. Therefore, the term “end mill” is generally used to refer to flat-bottomed cutters.)

In the end-mill process, a 1/4 in. end mill was bored down into the slab, forming a 1/4 in. hole. The bore was started in the middle and then worked outward by moving the mill in a helix until the specified outer dimension was achieved. In addition, the cutter was programmed to partially overlap the previous cut. This program was judged to be optimum for maintaining thickness uniformity.

Critical machine programming parameters were as follows:

- finished swatch thickness of 0.25 in.;
- disk diameter of 2.25 in.;
- well diameter of 1.25 in.; and
- well depth, leaving a uniform remaining wall thickness, as required (0.020, 0.040, 0.060, or 0.080 in.).

When the outside diameter was machined, about 0.010 in. of material was left to hold the swatch in place. The three swatches were separated from the slab by using a razor knife to cut them free.

Given that three swatches were milled from one pass, it might be expected that the thickness profiles of swatch numbers 1, 2, and 3 would be similar, and likewise for the next set of three swatches. Other factors that may have affected the dimensions included slight spot-to-spot variations in the density of the original block and stress built into the block.

The original swatch numbers corresponded to the sequence in which they were produced. The exceptions were those swatches with higher thickness variability that were replaced; each of these was given the number of the swatch it replaced. These replacements are noted in the thickness measurements.

Thickness measurements were performed onsite after milling to monitor thickness uniformity. Thicknesses were measured using an NSK model PF454 0 to 1 in. micrometer, calibrated with a Mitutoyo BE1-9-3 gauge block set (Mitutoyo America Corporation; Aurora, IL). Measurements for each swatch were recorded on a spreadsheet.

Photographs of the milled permeation specimens are presented in Figures 2 and 3. The specimen in Figure 2 was photographed from directly above the swatch; the 12 o'clock position is marked. The chemical permeant was spiked directly into the well. The second photograph is a side view, with a focus on the inside edge of the well.

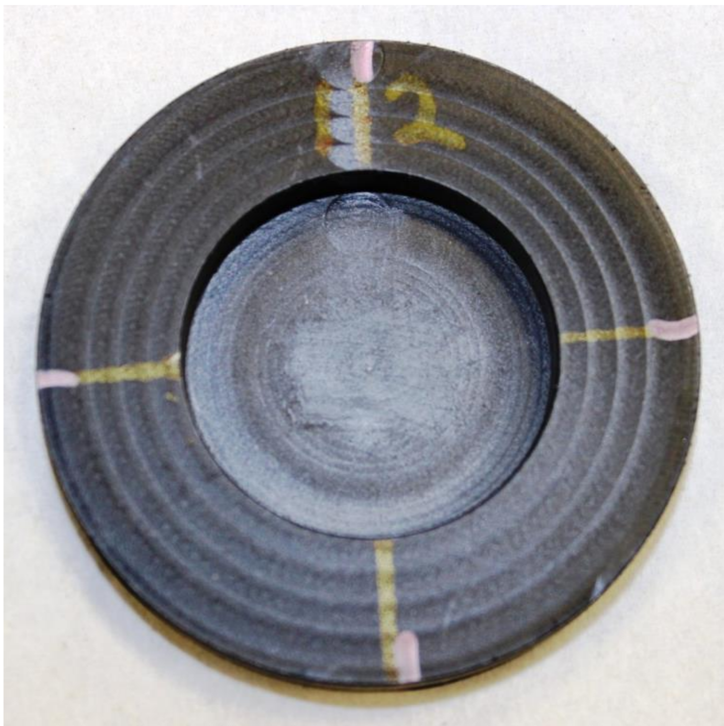

Figure 2. Photograph of an HDPE permeation specimen with an end-mill-machined well to contain chemical permeant (top view; 12 o'clock is marked).

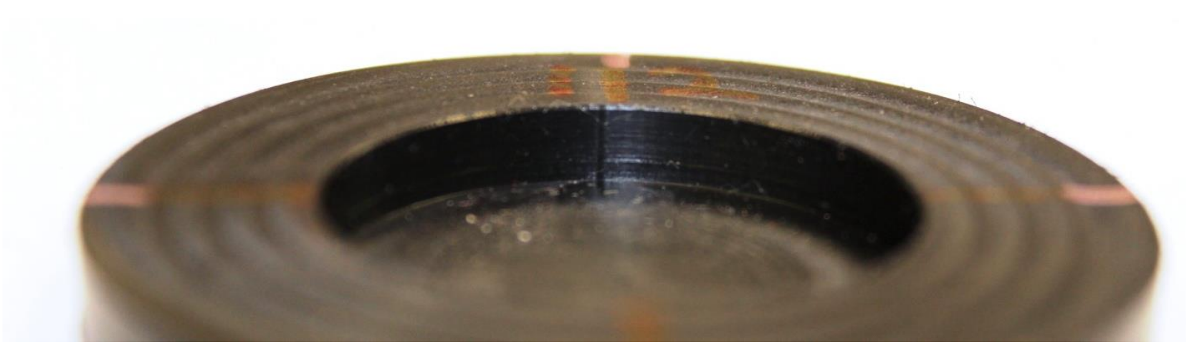

Figure 3. Photograph of an HDPE permeation specimen with an end-mill-machined well to contain chemical permeant (side view with focus on interior edge).

2.4 Procedure for Thickness Measurement as a Function of Position on the Swatch

The thickness was measured as a function of position on the well bottom. In Table 2, “clock position” refers to the location of the micrometer relative to the swatch, using a clock face as a reference. The bolt hole is positioned at 12 o’clock. The micrometer has a footprint diameter of about 6 mm. The first 6 mm is the location closest to the outside edge; therefore, three overlapping measurements could be made from the outside toward the inside. The C-clamp dimension did not allow measurements to be made closer to the swatch middle. Because measurements were made from both directions (from 12 and 6 o’clock), the middle 9–10 mm could not be measured with a standard micrometer.

To measure the center of the permeation specimen, a specialized micrometer was required. The swatches were taken to an L&W micrometer (model 1-2.08; Lorentzen & Wettre; Kista, Sweden) operated by M. Win (RDCB-DET-T) of the U.S. Army Edgewood Chemical Biological Center, Engineering Directorate; Test, Reliability, and Evaluation Branch. This micrometer provided readings to 0.1 mil; however, the measuring anvil was about 1.78 cm in diameter. Therefore, the measurement area covered the middle 50% of the diameter, and only the highest point within that area was determined.

The measurement resolution is 0.1 mil for the Scherr–Tumico (S&T) micrometer (S-T Industries; St. James, MN) and 0.5 mil for the Mitutoya micrometer. The values were reported to 0.01 mil to allow printing of the statistics. The two micrometers were also compared after calibration (as shown in the last four columns of Table 2). Calibration was performed with a Mitutoyo Corporation gauge block set, either identification no. 1101544 (calibration valid until 12 February 2014) or model BE1-10T-0A/A (grade 0), serial no. 1101545.

3. RESULTS

3.1 Measured Thickness Values Supporting Development of Method for Production of Permeation Specimens

The thickness values are reported in Table 2 for both the no-lube and lube-cooled swatches. For example, for the 1 o’clock position at the measurement closest to the outside rim, the value was 8.7 mil. The corresponding measurement, still at 1 o’clock, about 6 mm further toward the inside, was 8.39 or 8.4 mil.

The statistics are included at the bottom of Table 2. The means for the lube-cooled measurements ranged from 8.7 to 9 mil, depending on the micrometer. The no-lube swatch had a mean thickness of 9.2–9.3 mil. The 95% confidence interval (CI) ranged from 0.03 to 0.05 mil for swatches with no-lube milling and from 0.09 to 0.11 mil for the lube-cooled swatches.

The no-lube technique was requested as an option to avoid sorption of nonpolar chemicals by the nonpolar HDPE, which can pre-swell a polymer and increase permeation of the test chemical. Using the no-lube technique produced a swatch with better thickness control. The no-lube technique also produced a swatch thickness that was higher than targeted; however, reproducibility of thickness within a swatch and among different swatches was more important. Therefore, the no-lube technique is recommended.

Table 2. Summary of Thickness Measurements*

Clock Position		**Thickness Measurement**							
		S&T		**Mitutoya**		**Difference: S&T–Mitutoya**			
Measuring Anvil, 6 mm		**Lube Cooled (mil)**	**No Lube (mil)**	**Lube Cooled (mil)**	**No Lube (mil)**	**Lube Cooled (mil)**	**Lube Cooled (%)**	**No Lube (mil)**	**No Lube (%)**
First	1	8.70	8.80	8.89	9.19	–0.193	–2.174	–0.387	8.890
	2	8.49	9.00	8.74	9.24	–0.250	–2.856	–0.232	8.742
	3	8.59	9.11	8.89	9.19	–0.296	–3.324	–0.080	8.890
	4	8.70	9.22	8.94	9.24	–0.243	–2.714	–0.017	8.939
	5	8.80	9.21	9.14	9.24	–0.338	–3.698	–0.027	9.137
	6	8.70	9.21	8.94	9.28	–0.243	–2.714	–0.077	8.939
	7	8.90	9.11	9.14	9.28	–0.236	–2.578	–0.179	9.137
	8	8.90	9.11	9.14	9.33	–0.236	–2.578	–0.228	9.137
	9	8.70	9.11	8.89	9.24	–0.193	–2.174	–0.130	8.890
	10	8.39	9.00	8.69	9.19	–0.303	–3.481	–0.183	8.692
	11	8.49	9.11	8.74	9.24	–0.250	–2.856	–0.130	8.742
	12 (bolt)	8.59	9.00	8.59	9.19	0.001	0.009	–0.183	8.593
Second	1	8.39	9.11	8.84	9.28	–0.451	–5.099	–0.179	8.840
	2	8.29	9.00	8.69	9.28	–0.405	–4.658	–0.281	8.692
	3	8.49	9.22	8.79	9.33	–0.299	–3.402	–0.116	8.791
	4	8.70	9.21	8.84	9.28	–0.144	–1.627	–0.077	8.840
	5	8.80	9.21	8.99	9.28	–0.190	–2.110	–0.077	8.989
	6	8.80	9.31	9.19	9.28	–0.387	–4.215	0.026	9.186
	7	8.70	9.21	8.89	9.33	–0.193	–2.174	–0.126	8.890
	8	8.70	9.21	8.99	9.33	–0.292	–3.249	–0.126	8.989
	9	8.59	9.22	8.79	9.28	–0.197	–2.238	–0.067	8.791
	10	8.39	9.21	8.59	9.28	–0.204	–2.372	–0.077	8.593
	11	8.39	9.21	8.59	9.28	–0.204	–2.372	–0.077	8.593
	12 (bolt)	8.49	9.21	8.74	9.33	–0.250	–2.856	–0.126	8.742
Third	1	9.11	9.23	9.24	9.33	–0.130	–1.404	–0.106	9.235
	2	9.21	9.41	9.43	9.48	–0.225	–2.384	–0.070	9.433
	3	9.31	9.21	9.53	9.48	–0.221	–2.323	–0.274	9.532
	4	9.31	9.52	9.48	9.43	–0.172	–1.814	0.082	9.482
	5	9.31	9.31	9.48	9.38	–0.172	–1.814	–0.073	9.482
	6	9.31	9.31	9.48	9.43	–0.172	–1.814	–0.123	9.482
	7	9.21	9.31	9.38	9.33	–0.176	–1.871	–0.024	9.384
	8	9.11	9.41	8.89	9.38	0.216	2.430	0.029	8.890
	9	8.90	9.21	9.04	9.38	–0.137	–1.513	–0.176	9.038
	10	8.39	9.41	9.09	9.33	–0.698	–7.678	0.078	9.087
	11	8.49	9.41	8.74	9.53	–0.250	–2.856	–0.119	8.742
	12 (bolt)	8.49	9.21	9.24	9.48	–0.74	–8.05	–0.27	9.24
Mean		8.74	9.20	8.99	9.32	–0.25	–2.74	–0.12	–1.255
Standard error		0.052	0.024	0.046	0.015	0.027	0.297	0.017	0.181
Median		8.697	9.208	8.914	9.285	–0.230	–2.481	–0.117	–1.246
Mode		8.697	9.208	8.890	9.285	–0.250	–2.856	–0.077	–0.827
Standard deviation		0.310	0.141	0.277	0.090	0.161	1.780	0.101	1.087
Sample variance		0.096	0.020	0.077	0.008	0.026	3.170	0.010	1.181
Range		1.023	0.716	0.938	0.346	0.960	10.481	0.469	5.085
Minimum		8.287	8.799	8.593	9.186	–0.744	–8.051	–0.387	–4.215
Maximum		9.310	9.515	9.532	9.532	0.216	2.430	0.082	0.869
95.0% Confidence level		0.105	0.048	0.094	0.030	0.055	0.602	0.034	0.368

*n = 36 measurements for each caliper-lube category. Thickness measurements are shown as a function of position around the diameter (clock position) and concentric diameters around the swatches. Two micrometers (S&T and Mitutoya) were used to compare no-lube and lube-cooled swatches.

The pattern of thickness values versus position shows that the swatch became thicker toward the inner center. This can be observed in Figures 4–7, which provide a plot of clock position versus thickness. All of the *y* axis spans were scaled over 1.2 mil so that the relative magnitudes can be visually compared. The 1st, 2nd, and 3rd labels refer to the concentric circles, from closest to the outside rim toward the center. The 1st and 2nd concentric circles have lower thickness values than the 3rd circle. In addition, there appears to be a pattern of thicker values between positions 2 and 6, especially for the lube-cooled swatch. Polynomial regression fits (5th order) are provided to visualize the data from the same concentric circle. Overall, one can see that the no-lube specimens had narrower thickness ranges within the permeation specimen.

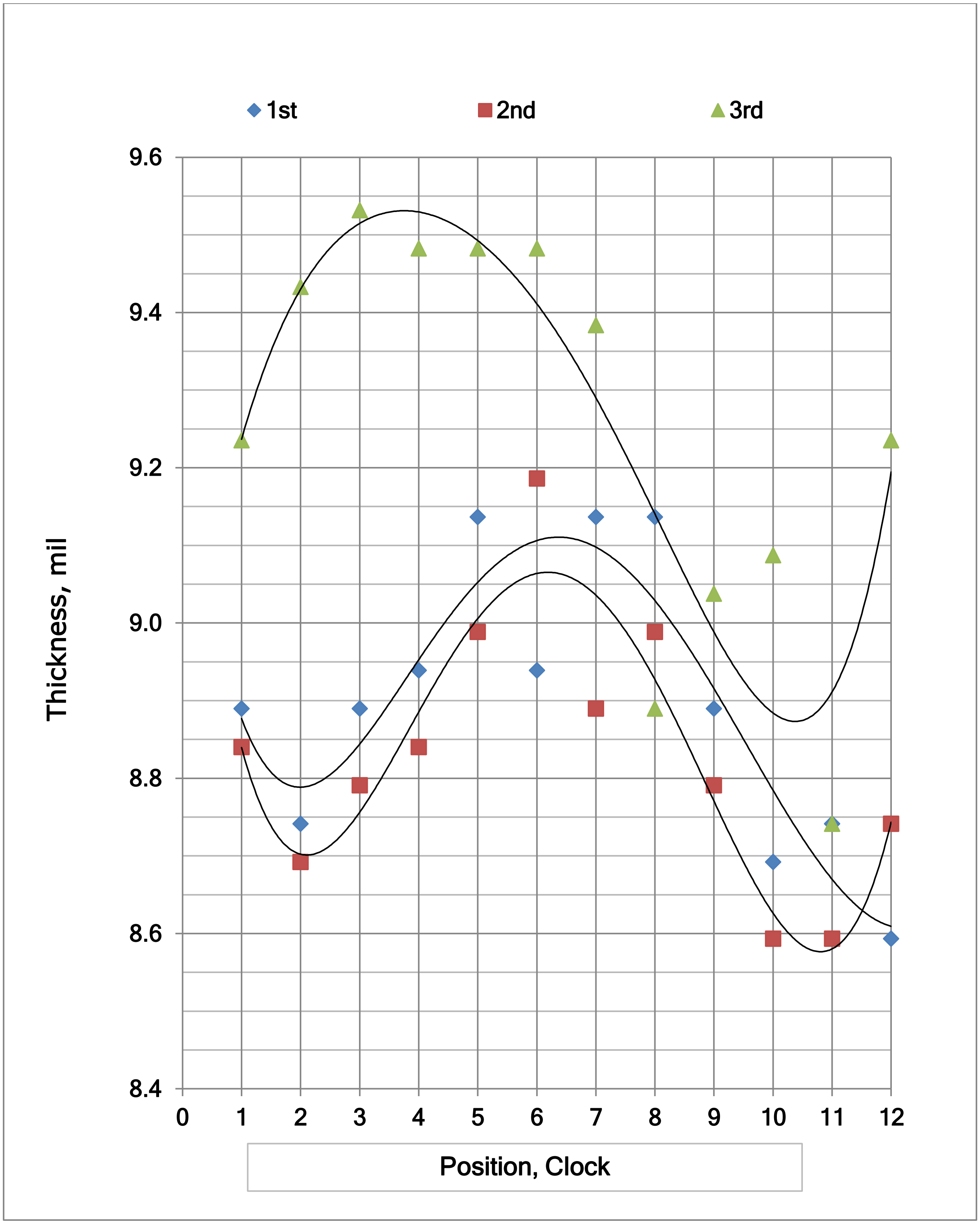

Figure 4. Corrected thickness vs position for lube-cooled swatches (Mitutoya micrometer).

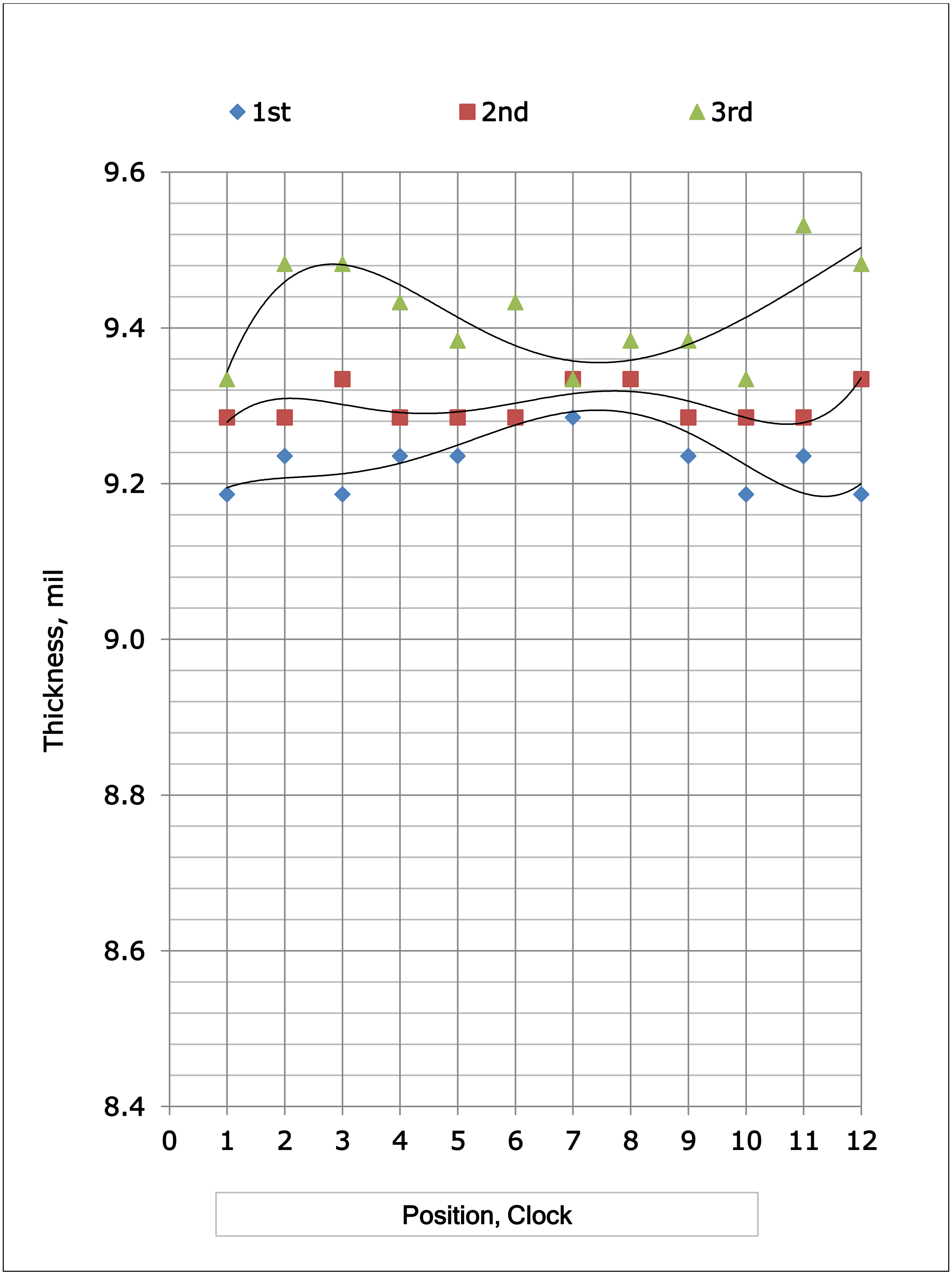

Figure 5. Corrected thickness vs position for no-lube swatches (Mitutoya micrometer).

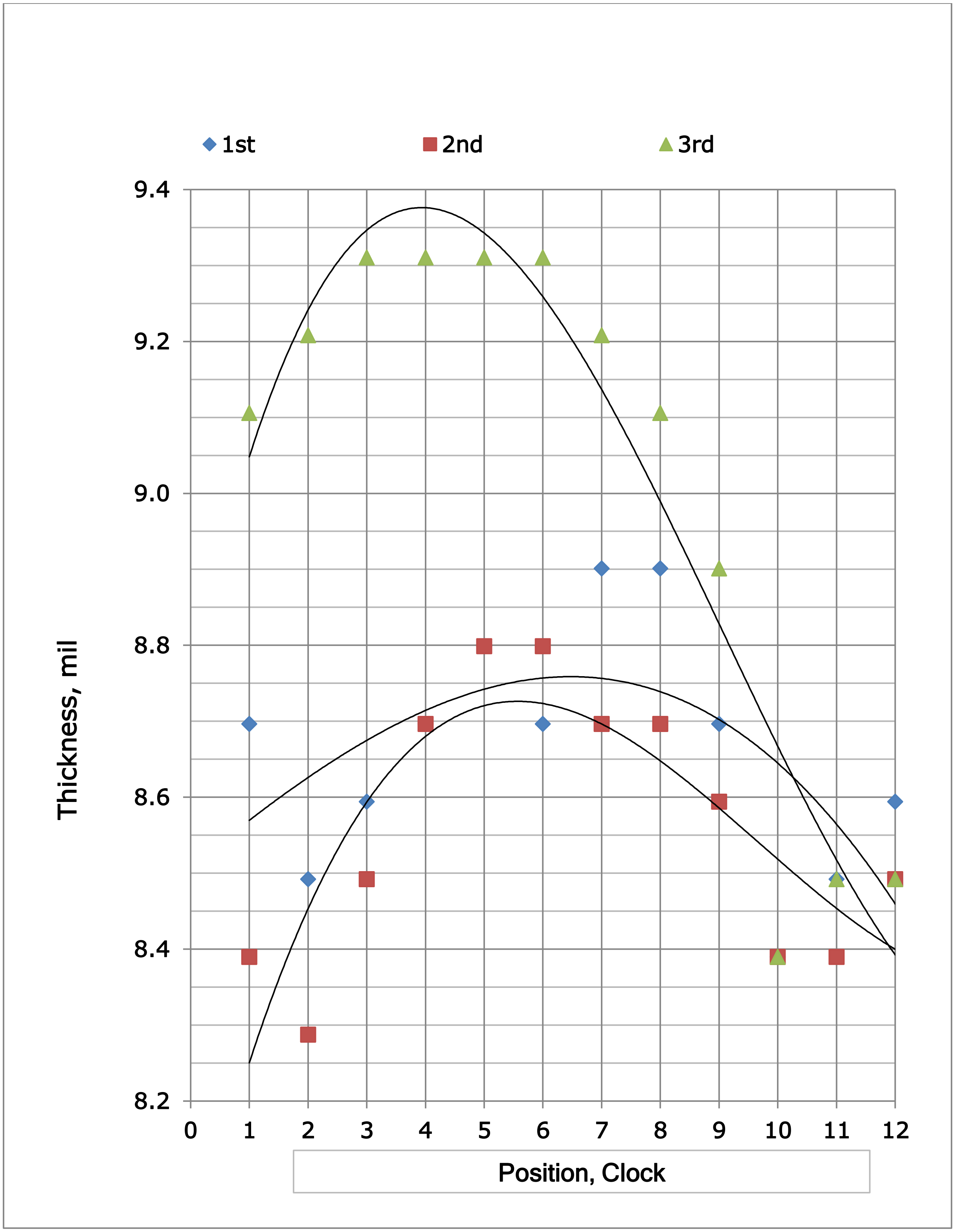

Figure 6. Corrected thickness vs position for lube-cooled swatches (S&T micrometer).

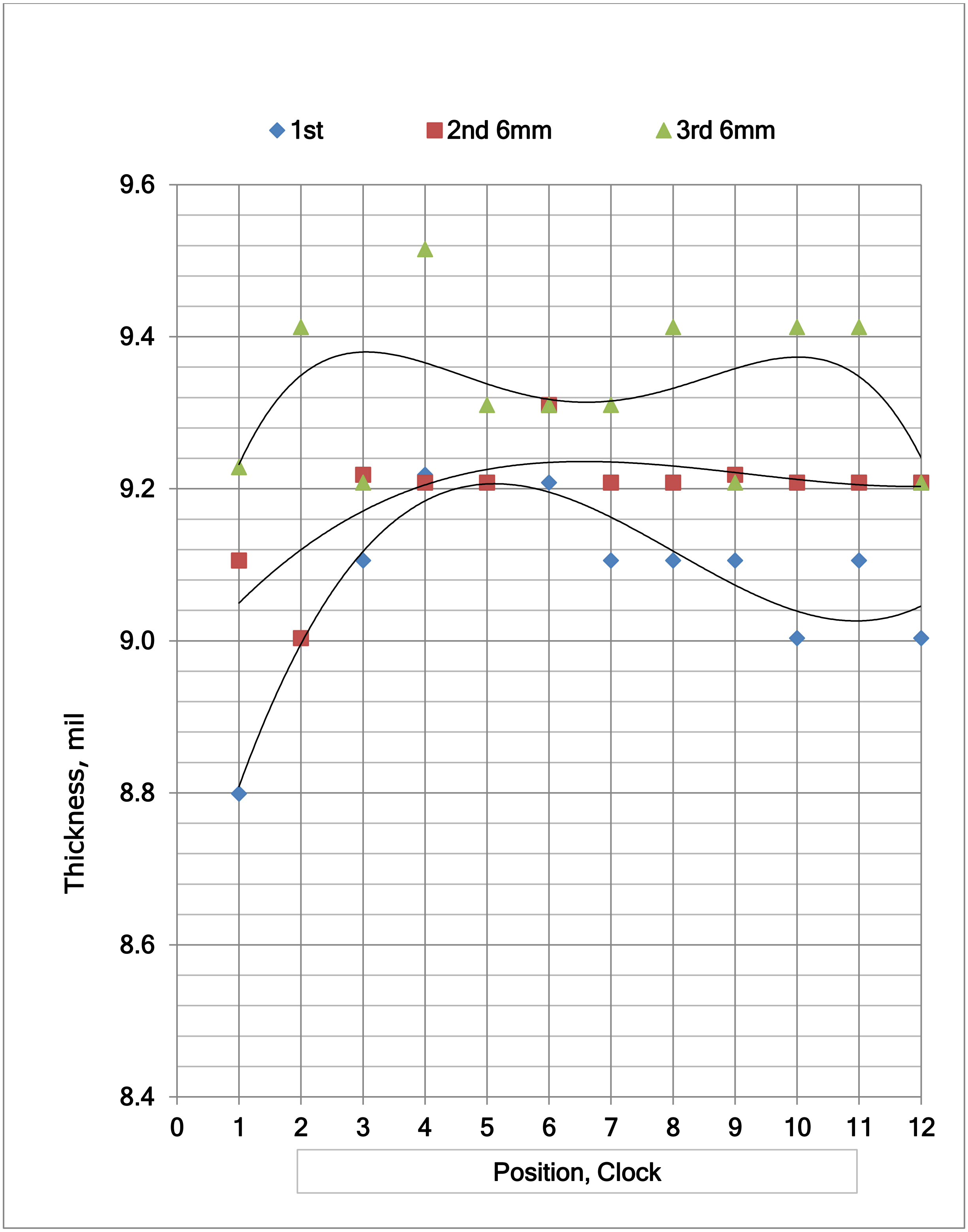

Figure 7. Corrected thickness vs position for no-lube swatches (S&T micrometer).

3.2 Thickness Comparison between Lube-Cooled and No-Lube Techniques: Non-Welded

The differences between the lube-cooled and no-lube swatch thickness values are compared in Table 3. All of the deviations were positive, with a couple of exceptions; therefore, the no-lube swatches were greater in thickness for the same target thickness. The values were 10.20 mil for the no-lube swatch and 9.60 mil for the lube-cooled swatch. These seem significantly greater than the micrometer measurements that were obtained toward the outside of the cup rim. The measurement for the no-lube swatch was 5.8% greater. This agrees with the outer rim measurements that were obtained using other micrometers, which were 3.5 to 5% greater.

Table 3. Thickness Comparison of Lube-Cooled and No-Lube Swatches
for a Cup Milled in a Swatch Composed of Continuum HDPE: Non-Welded

Thickness Difference: No Lube vs Lube Cooled			
S&T Micrometer		**Mitutoya Micrometer**	
mil	**%**	**mil**	**%**
0.1	1.2	0.3	3.2
0.5	5.7	0.5	5.3
0.5	5.6	0.3	3.2
0.5	5.7	0.3	3.2
0.4	4.4	0.1	1.1
0.5	5.6	0.3	3.7
0.2	2.2	0.1	1.6
0.2	2.2	0.2	2.1
0.4	4.5	0.3	3.7
0.6	6.8	0.5	5.4
0.6	6.7	0.5	5.3
0.4	4.5	0.6	6.5
0.7	7.9	0.4	4.8
0.7	8.0	0.6	6.4
0.7	7.9	0.5	5.8
0.5	5.6	0.4	4.8
0.4	4.4	0.3	3.2
0.5	5.5	0.1	1.1
0.5	5.6	0.4	4.8
0.5	5.6	0.3	3.7
0.6	6.8	0.5	5.3
0.8	8.9	0.7	7.4
0.8	8.9	0.7	7.4
0.7	7.8	0.6	6.3
0.1	1.3	0.1	1.1
0.2	2.2	0.0	0.5
–0.1	–1.1	0.0	–0.5
0.2	2.2	0.0	–0.5
0.0	0.0	–0.1	–1.1
0.0	0.0	0.0	–0.5
0.1	1.1	0.0	–0.5
0.3	3.3	0.5	5.3
0.3	3.3	0.3	3.7
1.0	10.9	0.2	2.6
0.9	9.8	0.8	8.3
0.7	7.8	0.2	2.6

The statistics for the thickness values obtained when using no-lube versus lube-cooled techniques are reported in Table 4. The mean thickness was 3.5 to 5% higher for the no-lube swatch; therefore, the target may have to be offset by this amount when the wells are machined. Note, the absolute values of the differences were employed in the statistical calculations.

Table 4. Summary Statistics for Thickness Comparison of Lube-Cooled and No-Lube Swatches for a Cup Milled in a Swatch Composed of Continuum HDPE: Non-Welded

Statistic*	**Thickness Difference: No Lube vs Lube Cooled**			
	S&T Micrometer		**Mitutoya Micrometer**	
	mil	**%**	**mil**	**%**
Mean	0.456	4.959	0.327	3.511
Standard error	0.046	0.495	0.040	0.424
Median	0.512	5.556	0.346	3.694
Mode	0.512	5.556	0.296	3.226
Standard deviation	0.275	2.973	0.237	2.541
Sample variance	0.076	8.837	0.056	6.459
Range	1.125	11.981	0.889	9.343
Minimum	–0.102	–1.111	–0.099	–1.053
Maximum	1.023	10.870	0.790	8.290
95.0% confidence level	0.093	1.006	0.080	0.860

*n = 36 measurements for each comparison condition.

Based on these results, all further machining of permeation specimens was performed using the end-mill method with the no-lube procedure. The overall summary and recommendations are found in Section 4.

3.3 Measured Thickness of Permeation Specimens: Non-Welded

The swatches were ranked by several thickness parameters, as shown in Tables 5–9. The thickness range (column three) was used to select swatches for permeation testing, with the 95% CI being used as a secondary parameter. The rankings were very similar, as expected. The swatch numbers in the range rank (second column) were selected for testing in the permeation cups: the lower the range, the higher the priority. The last four swatches with wide range values were set aside, for later testing with detector paper or for exploratory permeation tests. Later, the swatches were randomly selected for cell position within the permeation system. Table 9 lists specimens with poor variability for use in scouting experiments.

Table 5. HDPE Swatch Identification Number and Thickness Characterization for Within-Swatch Thickness Variability and Ranking for Permeation Testing: 20 mil Swatches, Non-Welded

Use Order (ID No.)	Range Rank (ID No.)	Thickness Range (mil)	95% Confidence Level (ID No.)	95% Confidence Interval (mil)	Minimum (ID No.)	Minimum (mil)	Mean (ID No.)	Mean (mil)
1	8	0.6	8	0.060	6	19.41	6	20.18
2	13	0.9	13	0.091	7	19.46	17	20.23
3	17	1	1	0.111	17	19.68	8	20.38
4	11	1.1	5	0.118	15	19.76	15	20.39
5	1	1.2	11	0.118	3	19.81	7	20.39
6	5	1.3	15	0.126	5	19.96	3	20.45
7	6	1.3	17	0.129	4	20.06	5	20.52
8	9	1.3	6	0.131	8	20.11	4	20.55
9	15	1.35	9	0.131	14	20.11	9	20.76
10	4	1.35	4	0.138	1	20.21	1	20.77
11	16	1.55	3	0.150	9	20.21	11	20.85
12	10	1.6	16	0.154	11	20.41	14	20.87
13	3	1.7	2	0.155	10	20.41	13	20.98
14	2	1.7	10	0.161	16	20.46	10	21.02
15	14	1.75	14	0.176	13	20.51	16	21.14
16	12	1.8	7	0.224	2	20.71	2	21.35
17	7	2.2	12	0.249	12	20.71	12	21.43

Table 6. HDPE Swatch Identification Number and Thickness Characterization for Within-Swatch Thickness Variability and Ranking for Permeation Testing: 40 mil Swatches, Non-Welded

Use Order (ID No.)	Range Rank (ID No.)	Thickness Range (mil)	95% Confidence Level (ID No.)	95% Confidence Interval (mil)	Minimum (ID No.)	Minimum (mil)	Mean (ID No.)	Mean (mil)
1	10	0.60	10	0.067	4	38.98	6	40.16
2	14	0.80	14	0.083	17	39.47	2	40.19
3	2	0.85	2	0.085	6	39.68	17	40.26
4	12	0.90	3	0.099	16	39.76	4	40.29
5	5	0.95	8	0.103	2	39.78	5	40.38
6	11	0.95	5	0.104	5	39.88	3	40.49
7	3	1.00	12	0.106	1	39.88	16	40.53
8	8	1.05	11	0.118	3	40.08	8	40.65
9	7	1.10	7	0.123	8	40.26	7	40.77
10	9	1.35	16	0.144	7	40.26	9	40.82
11	16	1.35	9	0.147	9	40.41	10	40.87
12	17	1.35	15	0.153	15	40.41	1	40.95
13	6	1.60	13	0.155	10	40.61	15	41.08
14	15	1.60	17	0.161	11	40.76	11	41.15
15	13	1.65	6	0.164	12	40.81	12	41.19
16	1	2.00	4	0.185	13	40.86	14	41.20
17	4	2.20	1	0.221	14	40.91	13	41.38

Table 7. Within-Swatch Thickness Variability for HDPE Swatches and Selection for Permeation Testing: 60 mil Swatches, Non-Welded

Use Order (ID No.)	Range Rank (ID No.)	Thickness Range (mil)	95% Confidence Level (ID No.)	95% Confidence Interval (mil)	Minimum (ID No.)	Minimum (mil)	Mean (ID No.)	Mean (mil)
1	9	0.9	9	0.091	8	60.25	8	60.76
2	10	0.9	10	0.094	12	60.35	4	60.90
3	14	0.9	6	0.111	7	60.4	9	60.99
4	6	1	14	0.112	4	60.45	10	61.01
5	13	1.1	13	0.113	9	60.5	13	61.10
6	5	1.15	1	0.119	10	60.5	6	61.13
7	1	1.2	3	0.127	15	60.5	12	61.13
8	3	1.2	5	0.128	13	60.55	11	61.16
9	8	1.2	12	0.129	16	60.55	5	61.19
10	11	1.25	4	0.129	6	60.65	15	61.19
11	4	1.3	8	0.134	3	60.75	7	61.26
12	15	1.35	11	0.136	11	60.75	16	61.31
13	2	1.4	2	0.139	5	60.8	14	61.36
14	12	1.45	15	0.149	14	60.9	3	61.37
15	16	1.75	16	0.172	2	61.05	2	61.53
16	7	2	7	0.206	1	61.3	1	61.73

Table 8. Within-Swatch Thickness Variability of HDPE Swatches and Selection for Permeation Testing: 80 mil Swatches, Non-Welded

Use Order (ID No.)	Range Rank (ID No.)	Thickness Range (mil)	95% Confidence Level (ID No.)	95% Confidence Interval (mil)	Minimum (ID No.)	Minimum (mil)	Mean (ID No.)	Mean (mil)
1	10	0.8	2	0.086	10	79.72	10	80.01
2	2	0.85	9	0.092	5	79.82	13	80.32
3	4	0.85	4	0.093	13	79.92	5	80.49
4	1	0.9	10	0.098	12	80.07	12	80.74
5	15	0.9	13	0.098	16	80.07	16	80.81
6	13	0.95	1	0.099	6	80.32	6	80.89
7	6	1	15	0.099	11	80.32	7	80.91
8	9	1	6	0.114	7	80.37	9	80.94
9	12	1.2	12	0.125	14	80.37	14	80.98
10	14	1.2	14	0.132	8	80.42	11	81.09
11	8	1.3	16	0.132	9	80.42	4	81.26
12	7	1.35	7	0.139	2	80.92	8	81.31
13	5	1.4	8	0.139	4	80.92	2	81.31
14	11	1.4	11	0.144	1	81.07	1	81.50
15	16	1.4	5	0.158	15	81.07	15	81.50
16	3	1.6	3	0.189	3	81.22	3	81.81

Table 9. Thickness Statistics for Extra HDPE Swatches with Poor Thickness Variability, for Use in Preliminary Experimentation: Non-Welded Swatches*

Statistic	Thickness (mil)				
	Extra 80-2	Extra 80-10	Extra 60-7	Extra 60-9	Extra 60
Mean	81.125	80.175	59.789	59.32	62.006
Standard error	0.25	0.069	0.16	0.22	0.10
Median	81.07	80.22	59.6	59.3	62
Mode	81.07	79.97	59.25	60	62
Standard deviation	0.75	0.21	0.49	0.67	0.31
Sample variance	0.57	0.047	0.24	0.45	0.096
Range	2	0.65	1.45	2.2	0.9
Minimum	80.07	79.87	59.25	57.8	61.6
Maximum	82.07	80.52	60.7	60	62.5
Sum	730.13	721.58	538.1	533.9	558.05
95.0% Confidence level	0.578	0.160	0.376	0.517	0.238

**n* = 9 extra HDPE swatches measured for each thickness set.

3.4 Plots of Specimen Measurement Position versus Thickness

The nature and extent of thickness variability within each permeation specimen can be better visualized by inspection of a plot. Figure 8 is a plot of measurements that are referenced using clock positions (1–12 o'clock) on the swatch disk. The outer circle is labeled with the specimen identification number: No. 10 for the specimen with the low 95% CI of 0.067 mil, and No. 15 for the specimen with the high 95% CI of 0.15 mil. The inner circle was labeled "mid". Specimen No. 10, with low CI, had thickness values between 40.1 and 41.2 mil. The outer and mid values overlapped at positions 1–5 o'clock, and the remainder were similar in thickness. The specimen with the higher CI had thickness values between 42.0 and 40.4 mil. The operation of the end mill appears to correlate with the thickness trend between the outer and mid positions, although they span a wide range. In ranking the specimens for use in testing, No. 15 ranked last and was not selected. No. 10 ranked fourth, based on range rather than 95% CI, and it was selected for permeation testing.

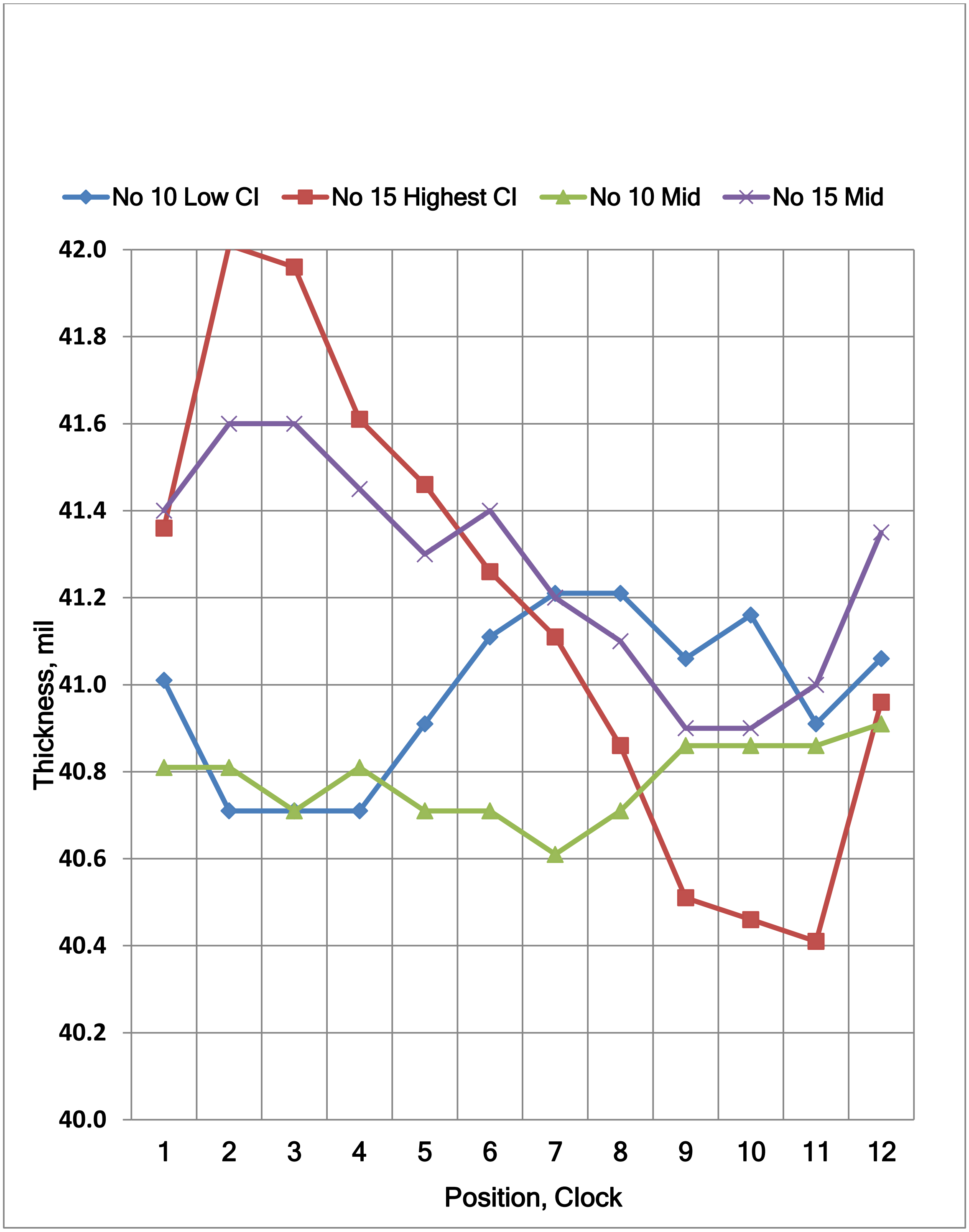

Figure 8. Thickness measurement location (clock position) vs thickness for non-welded swatches. Specimen No. 10 had a low 95% CI of 0.067 mil, and specimen No. 15 had a high 95% CI of 0.15 mil.

Table 10 provides a summary of the ranking of minimal thickness variability, based on range, for thickness sets of 20, 40, 60, and 80 mil. For a 12-cell permeation system, the least variable (from 1 to 12) specimens were randomly selected for the 12 cells.

Table 10. Within-Swatch Thickness Variability of HDPE Swatches for Range-Based Ranking and Selection for Permeation Testing: Non-Welded

Rank	**Range**			
	20 mil	**40 mil**	**60 mil**	**80 mil**
1	8	10	9	10
2	13	14	10	2
3	11	2	14	4
4	1	12	6	1
5	5	5	13	15
6	6	11	5	13
7	9	3	*1*	6
8	15	8	3	9
9	4	7	8	12
10	16	9	11	14
11	10	16	4	8
12	3	17	15	7
13	2	6	2	5
14	14	15	12	11
15	12	13	16	16
16	7	1	7	3

3.5 Measured Thickness of Permeation Specimens: Welded

The overall results for the welded permeation specimens are presented in Table 11. The swatches with the top 12 identification numbers exhibited less variability with respect to thickness range and were recommended for use in permeation testing. The bottom 5–8 identification numbers (shaded cells) represent extras that could be used for preliminary testing. The lower the placement on Table 11, the more variable the thickness; therefore, the bottom-numbered swatches should be used for the less-critical permeation tests. Detailed data on thickness measurements is documented in appendix Tables A-1 through A-4.

Table 11. Within-Swatch Thickness Variability of HDPE Swatches for Range-Based Ranking and Selection for Permeation Testing: Welded

Rank	Range			
	20 mil	**40 mil**	**60 mil**	**80 mil**
1	2	12	17	12
2	11	2	6	3
3	1	1	8	13
4	9	10	13	11
5	8	8	15	17
6	15	17	10	7
7	6	7	9	10
8	10	6	11	16
9	16	11	4	2
10	5	4	12	14
11	3	14	16	15
12	4	9	2	9
13	12	3	5	4
14	19	13	3	8
15	18	5	14	1
16	13	16	7	6
17	7	15	1	5
18	17	N/A	N/A	N/A
19	14	N/A	N/A	N/A
20	20	N/A	N/A	N/A

Note: shaded cells represent extra swatches that could be used for preliminary testing. N/A, not applicable.

The thickness measurements are presented in plots of measurement location (as clock position) versus thickness values in Figures 9–26. Several examples of permeation specimens of low, medium, and high variability are presented for the nominal 20 mil set. Two examples of low- and high-variability permeation specimens are presented for the 40, 60, and 80 mil sets. To facilitate visual comparison, the range on the *y* axis scales were set to be similar or identical for specimens with similar variability. Measurements of the center sector were added for some sets of specimens. The thickness versus location (clock position) appears to be correlated for the inner and outer sectors. The sectors deviate from this correlation for the higher variability permeation specimens. Most often, the outer sector exhibited the minimum thickness; therefore, this is where the initial breakthrough would be expected. Most of the thickness patterns showed a single minimum. Three permeation specimens displayed a double minimum, as shown in Figure 15 (specimen 40-2), Figure 19 (slight; specimen 60-6), and Figure 26 (specimen 80-8). Three of the specimens showed essentially no minima, as shown in Figure 20 (specimen 60-17), Figure 23 (specimen 80-3), and Figure 24 (specimen 80-12). These were all low-variability specimens. In several cases, the outer sector contributed the most to the increased variability.

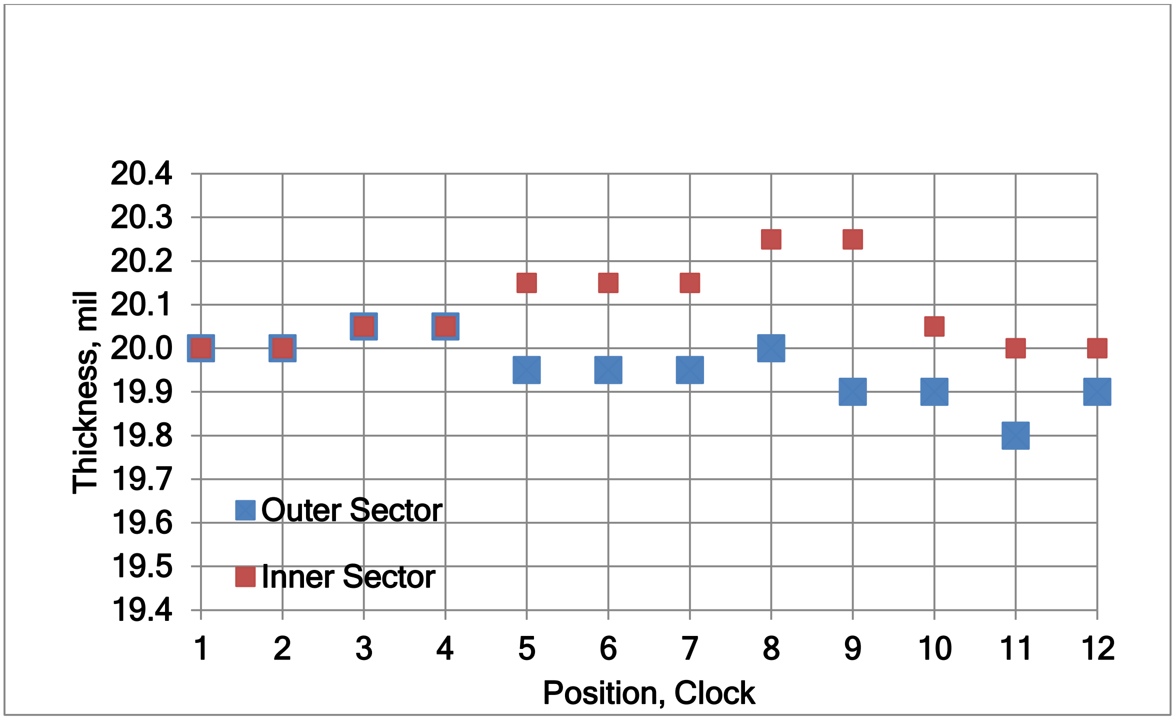

Figure 9. Permeation swatch location (as clock position) vs thickness for specimen 20-2: low variability, welded.

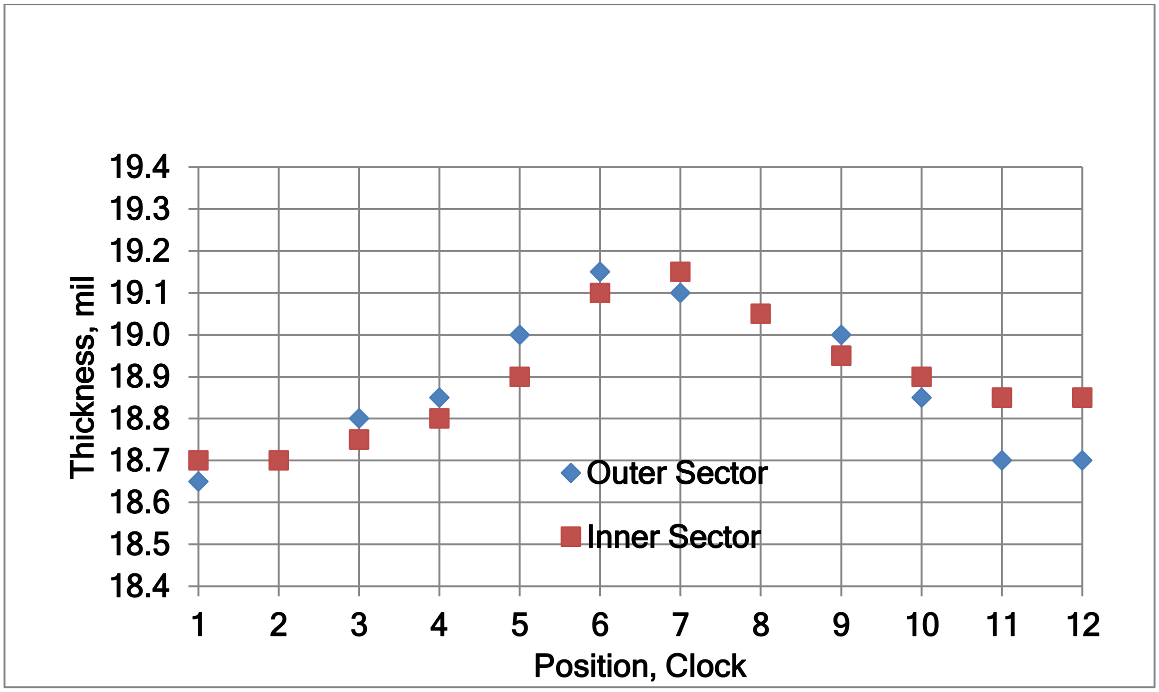

Figure 10. Permeation swatch location (as clock position) vs thickness for specimen 20-1: low variability, welded.

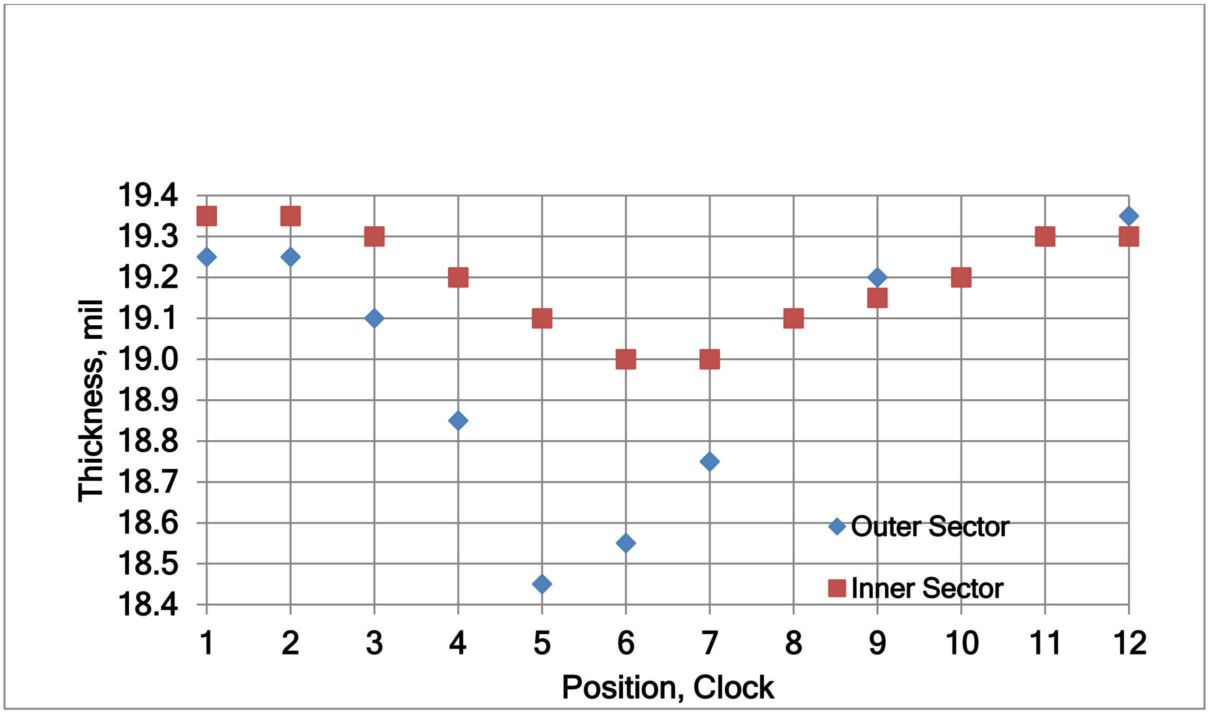

Figure 11. Permeation swatch location (as clock position) vs thickness for specimen 20-20: high variability, welded.

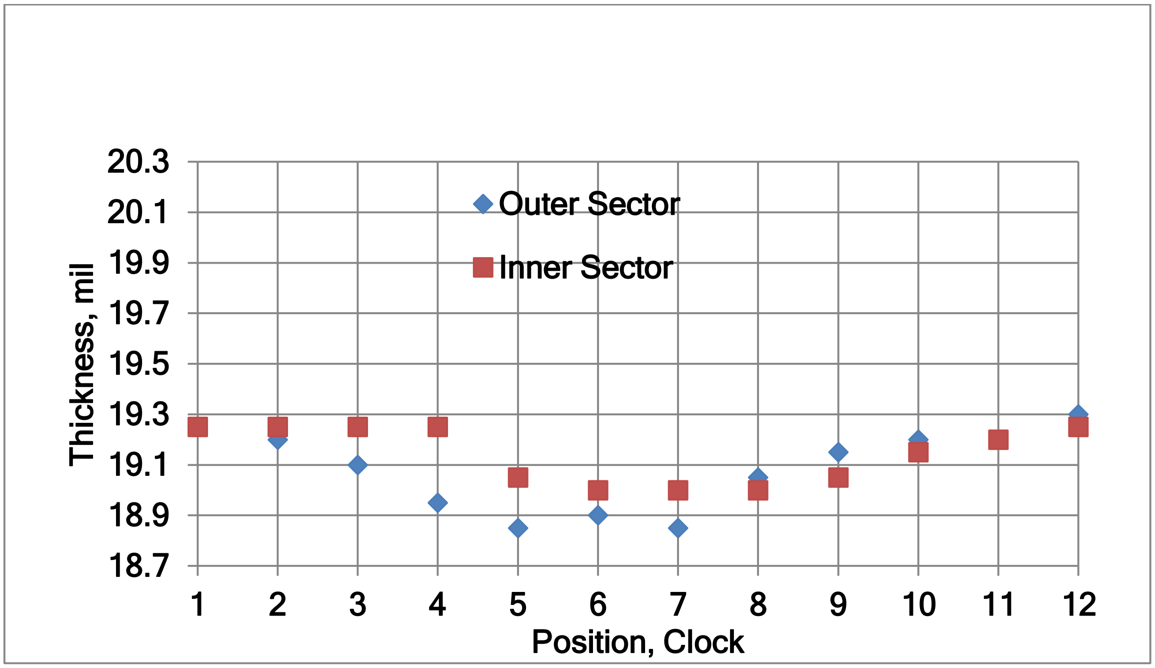

Figure 12. Permeation swatch location (as clock position) vs thickness for specimen 20-14: high variability, welded.

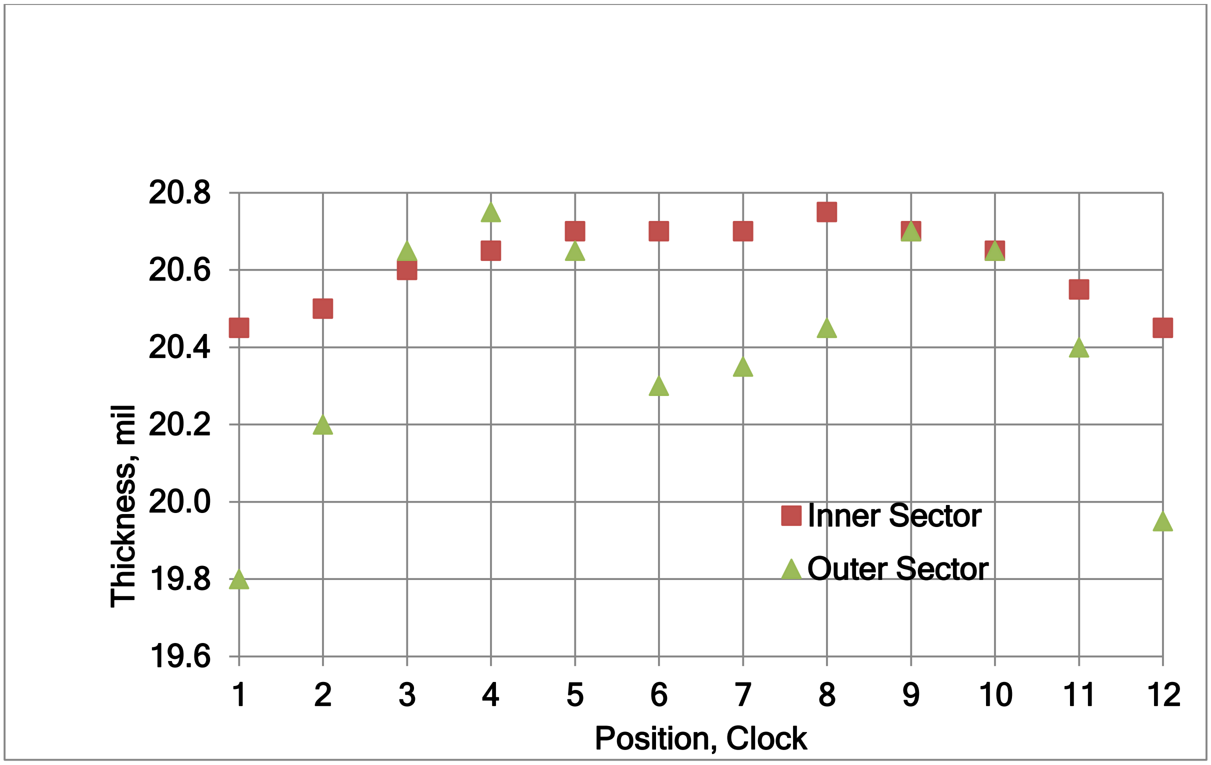

Figure 13. Permeation swatch location (as clock position) vs thickness for specimen 20-4: medium variability, welded.

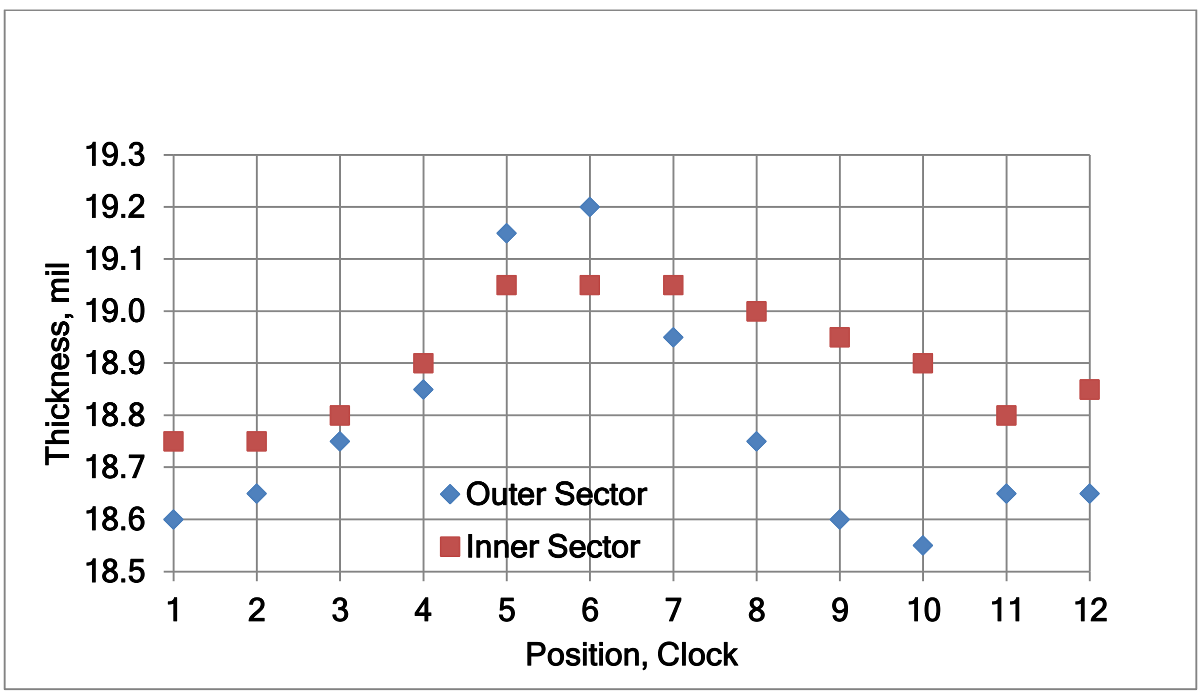

Figure 14. Permeation swatch location (as clock position) vs thickness for specimen 20-12: medium variability, welded.

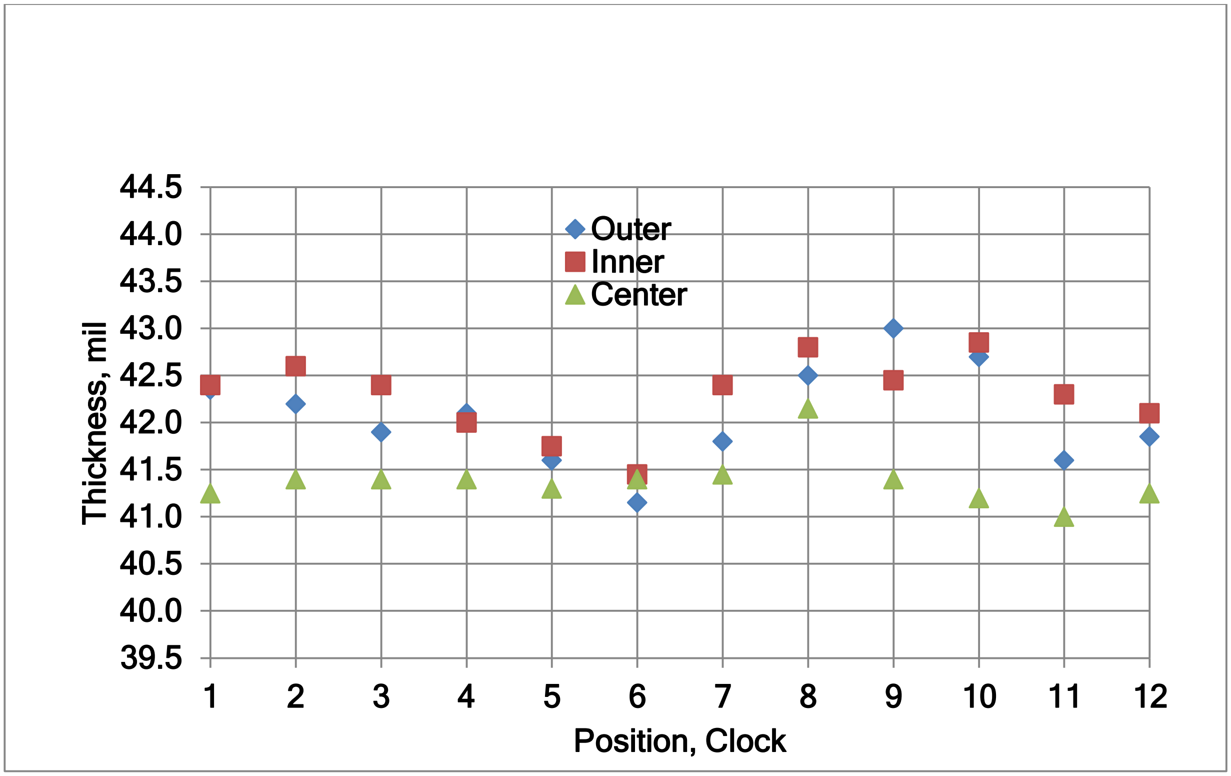

Figure 15. Permeation swatch location (as clock position) vs thickness for specimen 40-2: low variability, welded.

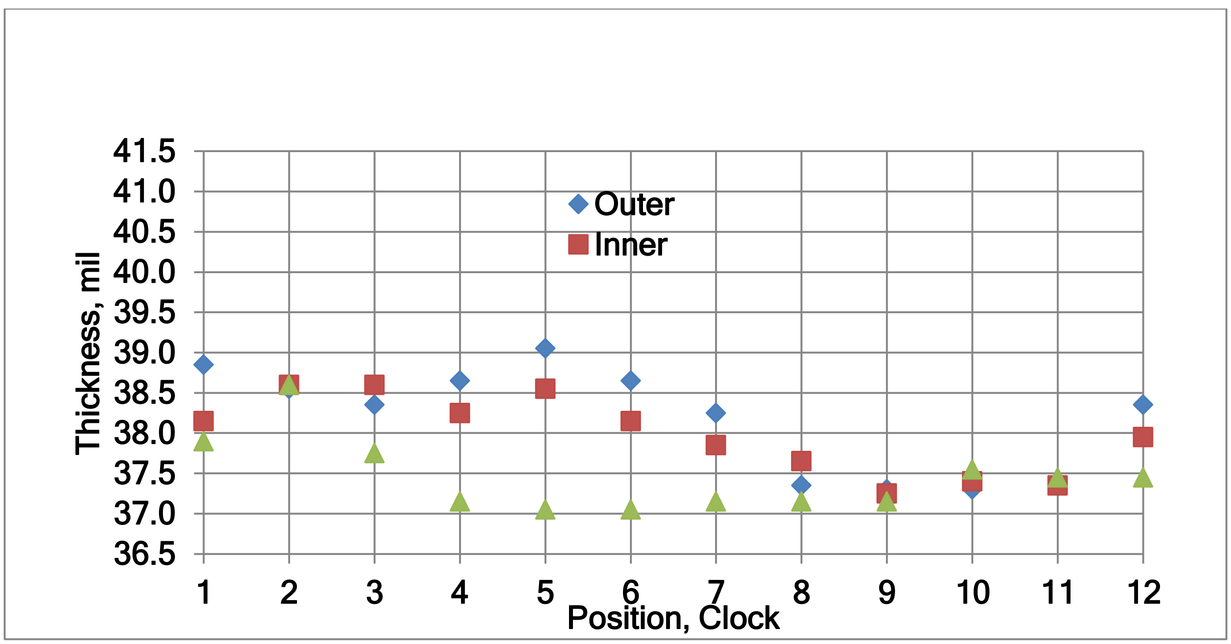

Figure 16. Permeation swatch location (as clock position) vs thickness for specimen 40-12: low variability, welded.

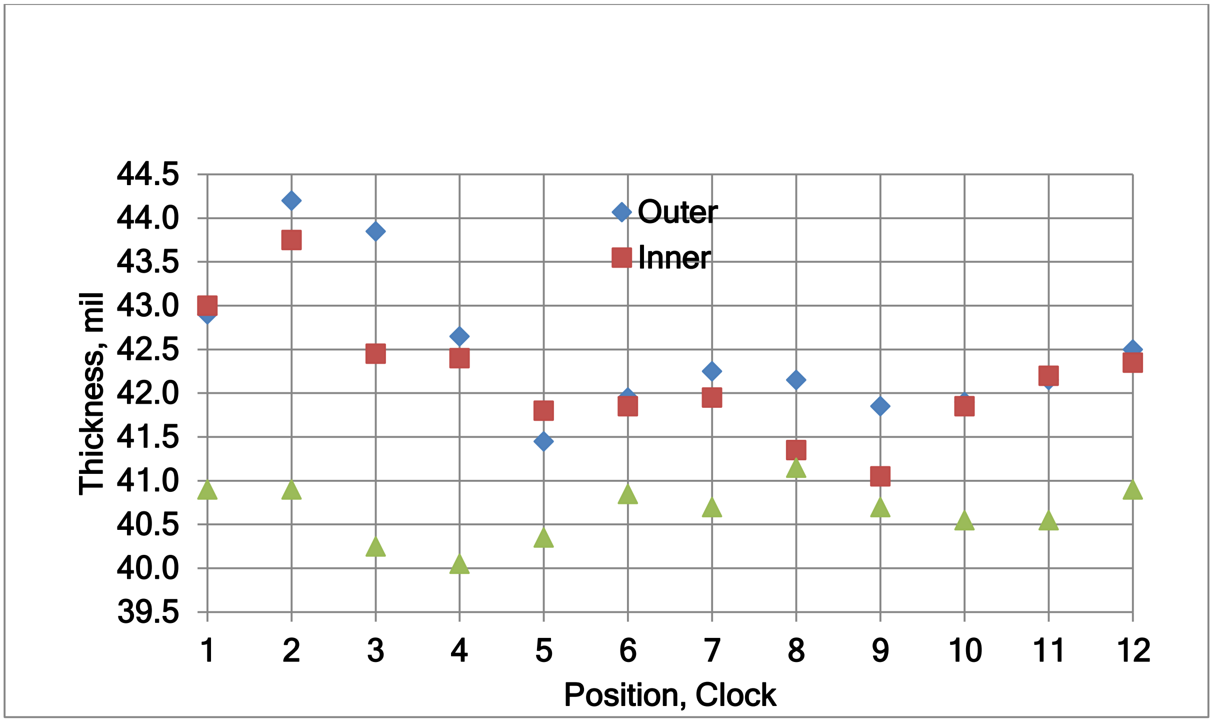

Figure 17. Permeation swatch location (as clock position) vs thickness for specimen 40-16: high variability, welded.

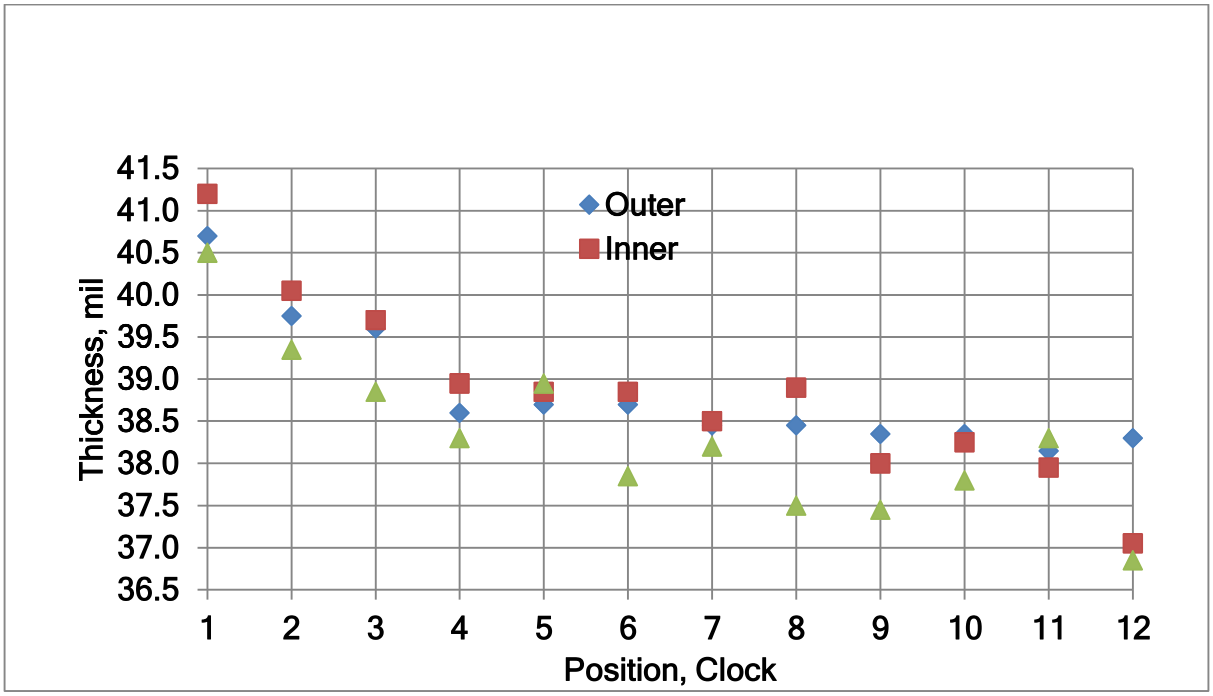

Figure 18. Permeation swatch location (as clock position) vs thickness for specimen 40-15: high variability, welded.

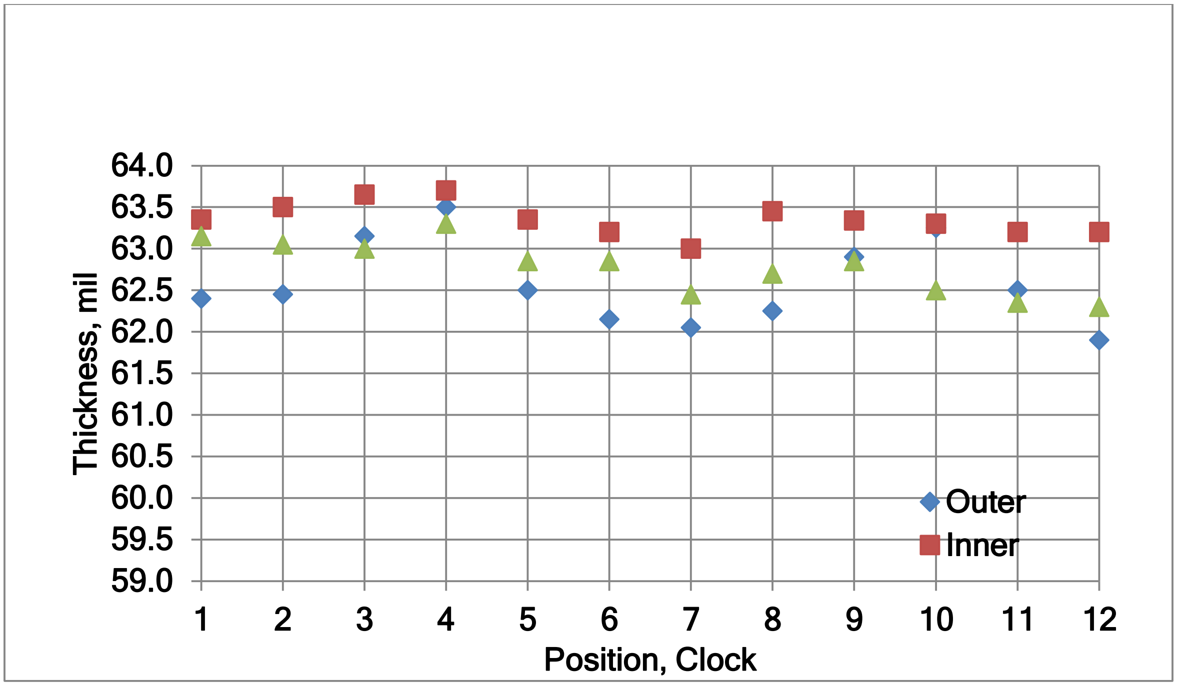

Figure 19. Permeation swatch location (as clock position) vs thickness for specimen 60-6: low variability, welded.

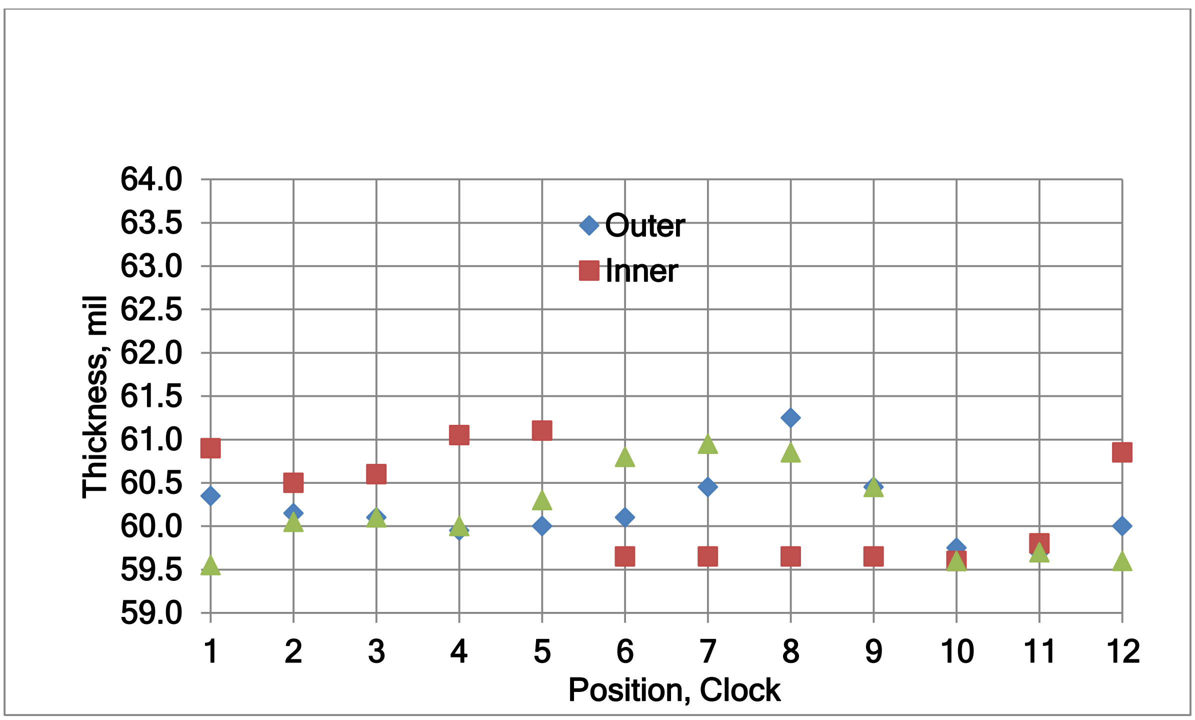

Figure 20. Permeation swatch location (as clock position) vs thickness for specimen 60-17: low variability, welded.

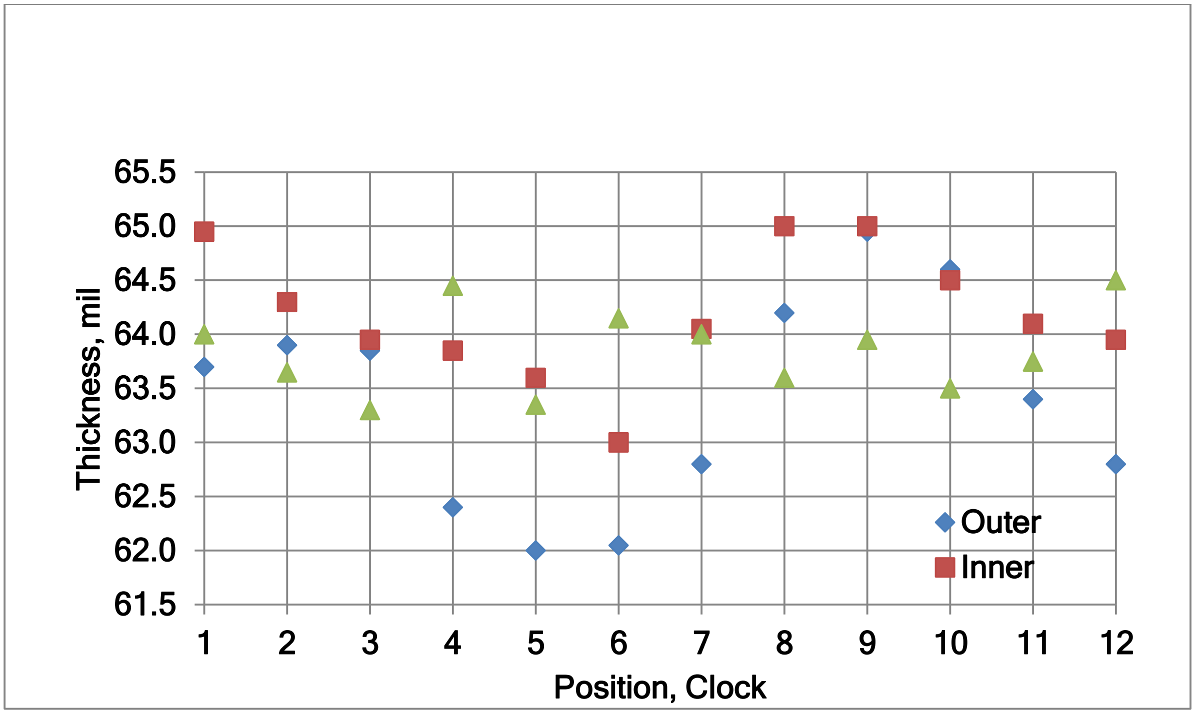

Figure 21. Permeation swatch location (as clock position) vs thickness for specimen 60-1: high variability, welded.

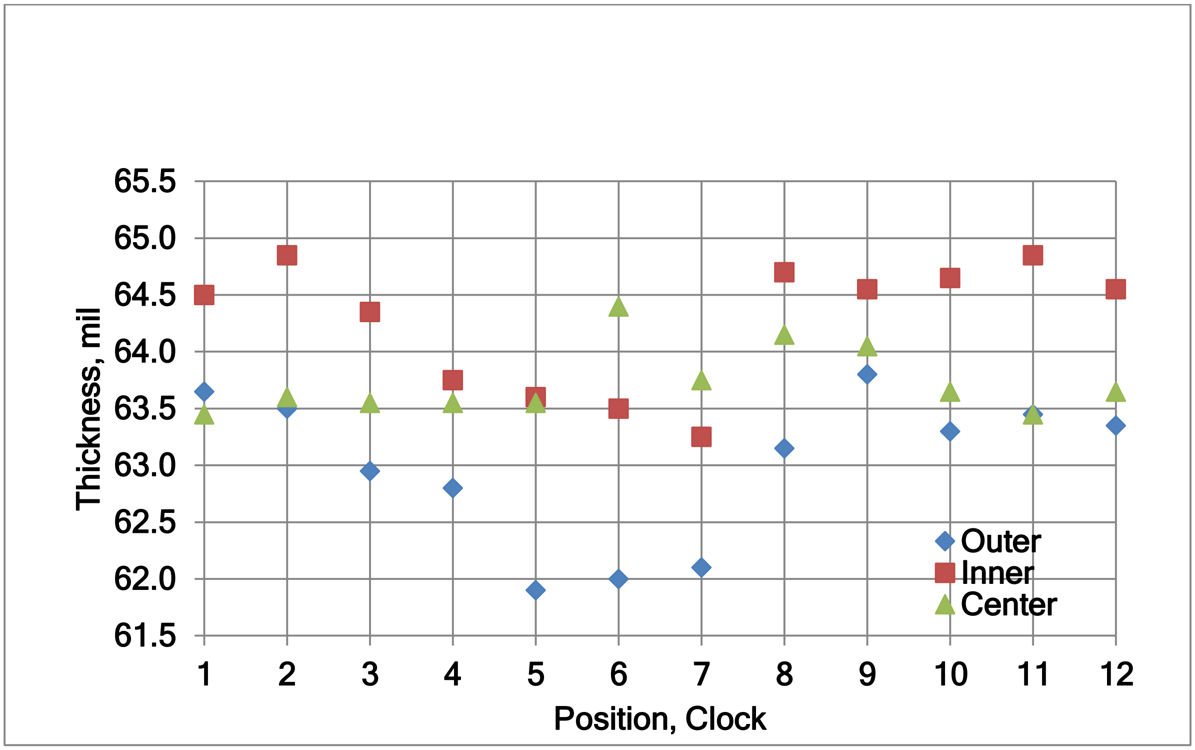

Figure 22. Permeation swatch location (as clock position) vs thickness for specimen 60-7: high variability, welded.

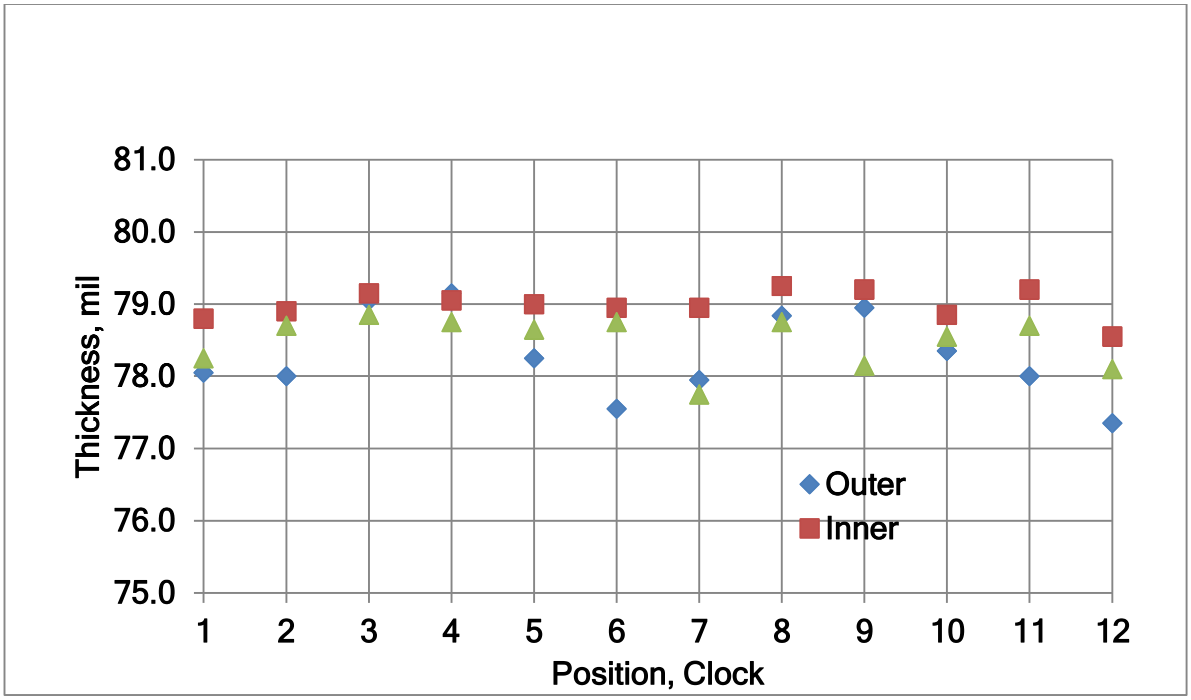

Figure 23. Permeation swatch location (as clock position) vs thickness for specimen 80-3: low variability, welded.

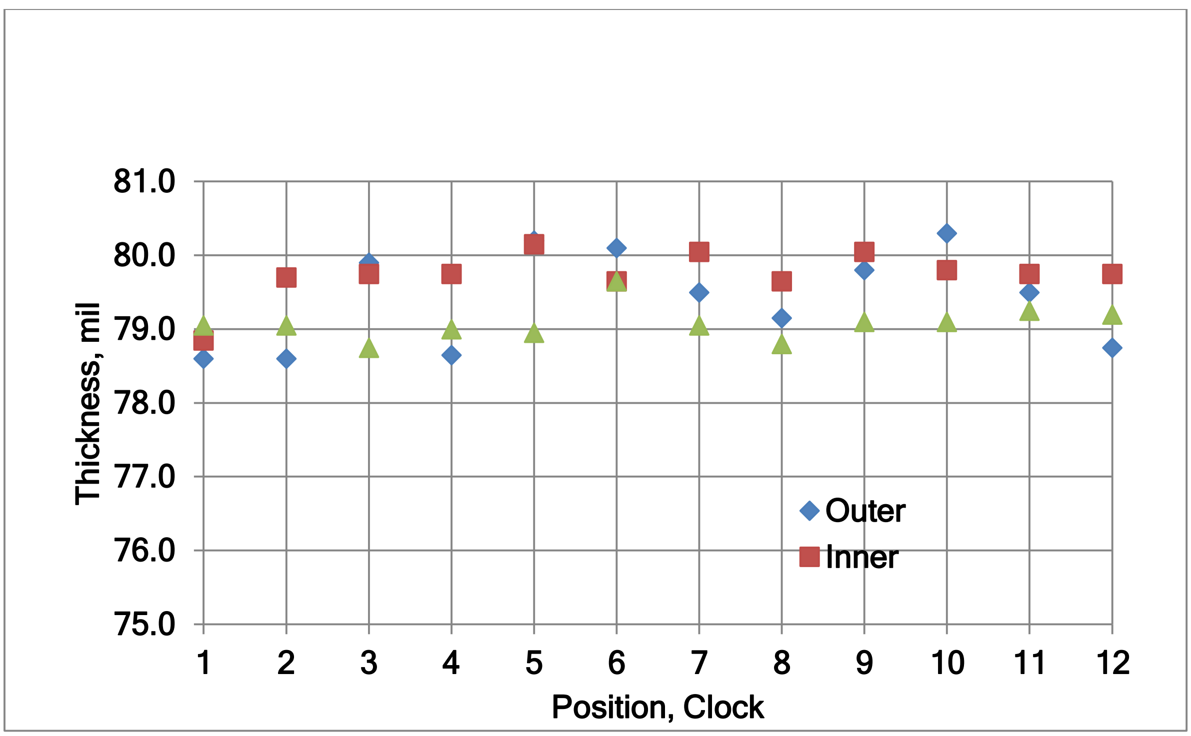

Figure 24. Permeation swatch location (as clock position) vs thickness for specimen 80-12: low variability, welded.

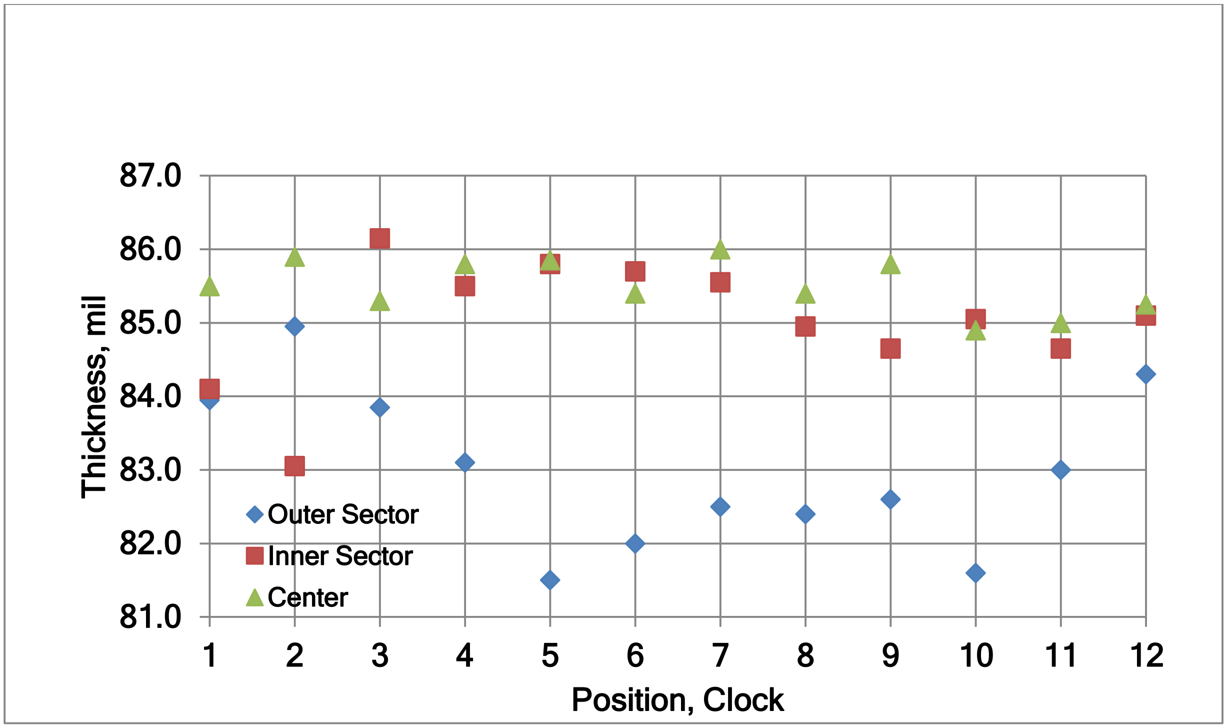

Figure 25. Permeation swatch location (as clock position) vs thickness for specimen 80-5: high variability, welded.

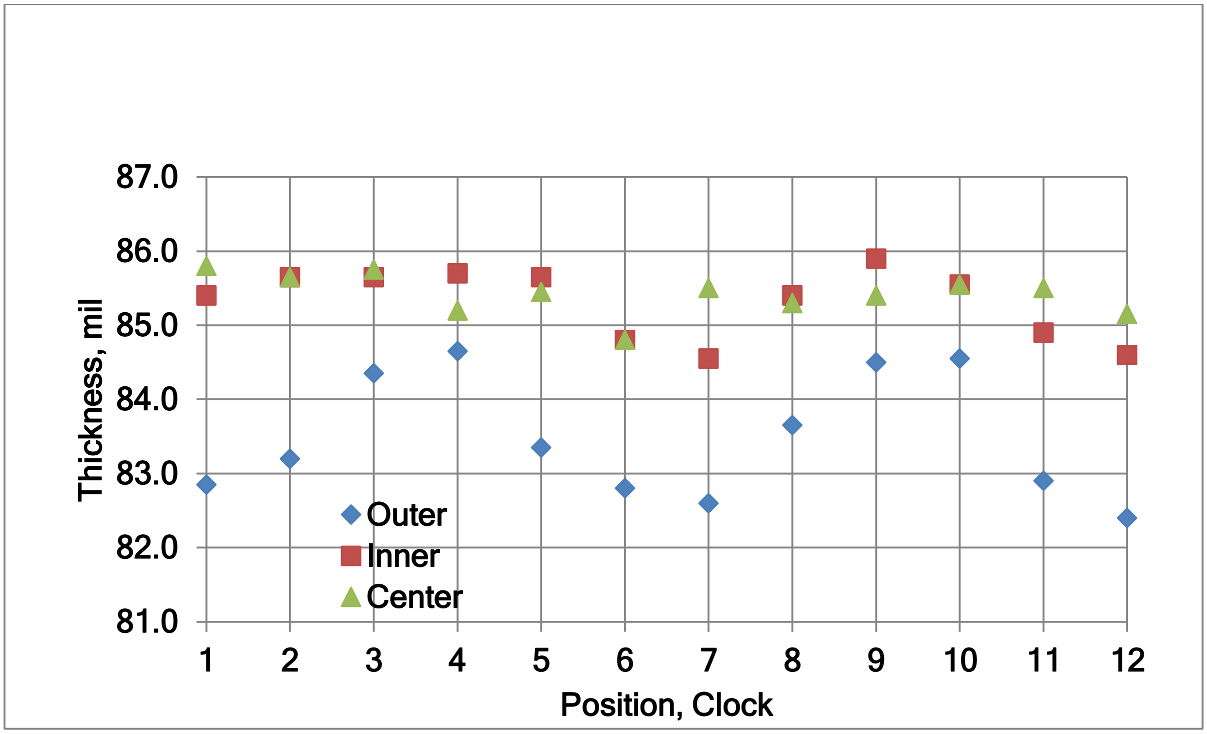

Figure 26. Permeation swatch location (as clock position) vs thickness for specimen 80-8: high variability, welded.

4. DISCUSSION AND SUMMARY

4.1 Permeation Specimen Production Recommendations

Based on the measurement results from the two swatches produced during development of the milling procedure, it was recommended that swatch production should be performed using the end-milling method. The no-lube technique is preferred based on the thickness variability and the ability to avoid contamination with a pre-swelling solvent.

The thinnest area of the swatch was near the rim. Breakthrough was likely to occur at these thinner areas. The swept area of the detection side of the swatch favored the mid area over the area near the rim. Therefore, the overall well diameter could be reduced from the current 35 mm to 30–32 mm. This would also keep the test liquid away from the waxed interface.

The following priorities should be specified to a milling facility. The consistency of thickness within each swatch is most important. The consistency of the thicknesses among the swatches is the next-most important consideration. Achieving a target thickness is less important.

The milling facility should also be requested to measure and report the thickness as a function of clock position at concentric circles within each swatch, as described in this report. These measurements should be used to monitor and adjust the machining process. The thermal-bonded blanks and non-welded material should be 3.5 in. square and 0.375–0.5 in. thick to fit a milling machine.

4.2 Variability of Swatch Thickness Compared with Target Specifications

The variability of the non-welded swatches is summarized in Table 12 and the variability of welded swatches is shown in Table 13. Both tables include the nominal swatch thicknesses and the number of replicates in the summary. The 16 replicates consisted of all of the swatches that were produced. The 12 swatch set consists of the best 12 swatches, based on a minimal range of thickness within each swatch. Those measurements that relate to the target specifications, that is, differences in thickness, are shown in bold italics.

With regard to the target specifications, none of the individual swatches had a within-swatch thickness range of 0.5 mil or less. The range for all nominal thickness values was a high of 1.6 mil (20 mil non-welded) to 2.2 mil (40 mil welded) over a 16 replicate set, and 1.35 mil (60/80 mil non-welded) to 1.7 mil (20 mil non-welded) over a 12 replicate set. The single swatch with the lowest range within one swatch was 0.6 mil (20 mil welded), which was above the 0.5 mil target. Regardless, this variability is consistent with that measured for the welded set and may be a characteristic limitation of the machining method.

With regard to the target specification that the mean thickness among the set of swatches be 1.2 mil or less, only the 60 mil non-welded set met this criterion for the 12 and 16 replicates.

Considering only the 12 replicates, the 20, 40, and 60 mil non-welded sets met the target, and the 80 mil set almost met the 1.2 mil range, with a value of 1.3 mil. This target specification is not as critical because the minimum individual swatch thickness versus breakthrough parameters can be plotted.

Inspection of the swatch variability of the range shows that there was not much difference among swatches ranked about 10th to 14th; therefore, selection of the best 12 would not be critical. However, the lowest ranking numbers, 15 and 16, often deviated considerably and should only be used for exploratory experimentation.

The results in Tables 12 (non-welded from IPS) and 13 (welded from SNL) can be compared to determine the degree of variability among a set of swatches at the two different specimen production sites. The 20 mil specimens produced at IPS had slightly greater variability, whereas for the remainder of the sets, the IPS-produced specimens had less variability, based on the range between highest and lowest values and the mean values.

Table 12. Summary of the Variability of Swatch Thickness by Range and Mean: Non-Welded from IPS

Thickness		**20 mil**		**40 mil**		**60 mil**		**80 mil**	
No. of Replicates		**16**	**12**	**16**	**12**	**16**	**12**	**16**	**12**
Range, within each swatch	Low (mil)	0.6	0.6	0.6	0.6	0.9	0.9	0.8	0.8
	High (mil)	2.2	1.7	2.2	1.6	2	1.35	1.6	1.35
	Difference (mil)	***1.6***	***0.9***	***1.6***	***1.0***	***1.1***	***0.45***	***0.8***	***0.55***
	Ratio	3.7×	2.8×	3.7×	2.7×	2.2×	1.5×	2×	1.7×
Mean, among a set of swatches	Low (mil)	20.2	20.2	40.16	40.16	60.67	60.76	80.0	80.
	High (mil)	21.4	20.98	41.38	41.08	61.73	61.31	81.8	81.3
	Difference (mil)	***1.25***	***0.8***	***1.22***	***0.92***	***0.97***	***0.55***	***1.8***	***1.3***
	% Range	6.2	4	3	2.3	1.6	0.9	2.3	1.6

Table 13. Summary of the Variability of Swatch Thickness by Range and Mean: Welded from SNL

Thickness		**20 mil**		**40 mil**		**60 mil**		**80 mil**	
No. of Replicates		**16**	**12**	**16**	**12**	**16**	**12**	**16**	**12**
Range, within each swatch	Low (mil)	0.5	0.5	2	2	1.7	1.7	2.1	2.1
	High (mil)	1.7	1.1	4.2	2.8	2.95	2.75	4.7	3.1
	Difference (mil)	***1.2***	***0.6***	***2.2***	***0.8***	***1.25***	***1.05***	***2.6***	***1***
	Ratio	3.4×	2.2×	2.1×	1.4×	1.74×	1.62×	2.238×	1.48×
Mean, among a set of swatches	Low (mil)	18.7	18.7	37.08	37.08	59.2	59.2	77.72	77.72
	High (mil)	19.8	19.3	41.9	41.2	64.4	63.3	84.7	83.3
	Difference (mil)	***1.1***	***0.6***	***4.8***	***4.2***	***5.2***	***4.1***	***6.9***	***5.6***
	% Range	5.7	3.0	12.2	10.6	8.4	6.7	8.5	6.9

The rather wide distribution of thickness values for any given set demonstrated that it was important to rank and select the specimens with the lowest variability for use in the test matrix.

Overall, one could recommend the swatches be accepted for use in the permeation experimentation.

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ACRONYMS AND ABBREVIATIONS

CI	confidence interval
CWA	chemical warfare agents
HD	distilled mustard
HDPE	high-density polyethylene
IPS	Innovative Plastic Solutions
SNL	Sandia National Laboratories
S&T	Scherr–Tumico (micrometers)
UMSC	Universal Munitions Storage Container

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APPENDIX

STATISTICAL EVALUATION OF PERMEATION SPECIMEN THICKNESS MEASUREMENTS: WELDED PERMEATION SPECIMENS

The specimens can be ranked on the basis of several criteria: the variability, which is based on range or 95% confidence interval (CI), or the minimum or mean thickness of the spectrum of specimens. Tables A1–A4 document the descriptive statistics for the four sets of thickness measurements. The first column lists the rank of the specimens in terms of lowest to highest variability based on the range. The second and third columns list the mean thicknesses and standard errors. The actual values of the ranges (in mil) are provided in the fifth column, followed by the range percentages {[(range/mean) – 1] × 100}. These range percentages provide insight into the relative variability. The minimum thicknesses are listed in the sixth column; these are the thickness levels that control breakthrough time. The minimum thickness, as a percentage of the mean, is calculated in the seventh column, and is given as the percent above zero for ease of comparison with percent range. The maximum thickness and number of thickness measurements are listed in columns eight and nine. The 95% CI and the percent 95% CI are presented in the last two columns. The 95% CI trend correlates with the range very closely, and either could be employed for the ranking. Not all column values were calculated for all of the thickness series. All of the ranking metrics were evaluated, and the rankings based on range were chosen for the selection of permeation specimens.

Table A1. Statistical Evaluation of Permeation Specimen Thickness Measurements: 20 mil Set, Welded

20 mil Range Sort	**Mean (mil)**	**Standard Error (mil)**	**Range (mil)**	**Range (%)**	**Minimum (mil)**	**Min/Mean (%)**	**Maximum (mil)**	**Count**	**95% Confidence Level (%)**	**95% Confidence Interval (mil)**
2	20.03	0.024	0.50	2.5	19.8	1.2	20.3	25	0.049	0.25
11	19.01	0.032	0.60	3.2	18.75	1.4	19.35	25	0.067	0.35
1	18.90	0.034	0.60	3.2	18.65	1.3	19.25	25	0.071	0.38
9	19.18	0.032	0.75	3.9	18.85	1.7	19.6	25	0.066	0.34
8	18.93	0.034	0.75	4.0	18.6	1.7	19.35	25	0.070	0.37
15	19.27	0.042	0.80	4.2	18.9	1.9	19.7	25	0.087	0.45
6	19.13	0.037	0.85	4.4	18.85	1.5	19.7	25	0.075	0.39
10	18.97	0.038	0.90	4.7	18.7	1.4	19.6	25	0.079	0.42
16	18.70	0.048	0.90	4.8	18.3	2.1	19.2	25	0.100	0.53
5	19.05	0.040	0.95	5.0	18.6	2.4	19.55	25	0.083	0.43
3	19.00	0.046	1.00	5.3	18.55	2.3	19.55	25	0.094	0.49
4	20.52	0.050	1.05	5.1	19.8	3.5	20.85	25	0.104	0.51
12	18.87	0.047	1.05	5.6	18.55	1.7	19.6	25	0.096	0.51
19	19.43	0.048	1.10	5.7	19	2.2	20.1	25	0.100	0.51
18	19.26	0.044	1.10	5.7	18.65	3.2	19.75	25	0.092	0.48
13	19.20	0.061	1.10	5.7	18.55	3.4	19.65	25	0.126	0.65
7	19.52	0.044	1.15	5.9	19	2.7	20.15	25	0.092	0.47
17	19.22	0.046	1.20	6.2	18.9	1.7	20.1	25	0.095	0.49
14	19.15	0.048	1.25	6.5	18.85	1.6	20.1	25	0.099	0.52
20	19.16	0.066	1.80	9.4	18.45	3.7	20.25	25	0.136	0.71

Table A2. Statistical Evaluation of Permeation Specimen Thickness Measurements: 40 mil Set, Welded

40 mil Range Sort	Mean (mil)	Standard Error (mil)	Median (mil)	Mode (mil)	Range (%)	Minimum (mil)	Maximum (mil)	Count	95.0% Confidence Interval (mil)
12	37.87	0.102	37.8	37.2	2.0	37.1	39.1	36	0.21
2	41.91	0.093	41.9	41.4	2.0	41.0	43.0	36	0.19
1	37.99	0.110	37.8	37.8	2.1	37.1	39.2	36	0.22
10	41.24	0.106	41.4	40.6	2.2	40.3	42.5	36	0.21
8	41.16	0.100	41.2	41.8	2.3	40.0	42.3	36	0.20
17	38.54	0.107	38.5	39.1	2.3	37.6	39.9	36	0.22
7	41.32	0.100	41.3	41.7	2.3	40.2	42.6	36	0.20
6	38.94	0.116	39.0	39.3	2.4	37.8	40.2	36	0.24
11	41.30	0.103	41.3	41.2	2.6	40.2	42.8	36	0.21
4	37.91	0.117	38.0	38.2	2.7	36.8	39.5	36	0.24
14	39.94	0.107	40.1	40.6	2.7	38.8	41.5	36	0.22
9	37.08	0.111	37.1	37.4	2.8	35.8	38.6	36	0.22
3	40.78	0.130	40.8	41.2	2.9	39.4	42.2	36	0.26
13	39.14	0.151	39.3	40.1	2.9	37.7	40.6	36	0.31
5	43.38	0.130	43.0	42.8	3.1	42.6	45.7	36	0.26
16	41.77	0.172	41.9	41.9	4.2	40.1	44.2	36	0.35
15	38.67	0.160	38.5	38.3	4.4	36.9	41.2	36	0.32

Table A3. Statistical Evaluation of Permeation Specimen Thickness Measurements: 60 mil Set, Welded

60 mil Range Sort	Mean (mil)	Standard Error (mil)	Median (mil)	Mode (mil)	Range (%)	Minimum (mil)	Maximum (mil)	Count	95.0% Confidence Interval (mil)
17	60.20	0.09	60.1	59.7	1.7	59.55	61.25	36	0.17
6	62.91	0.08	63.0	62.5	1.8	61.9	63.7	36	0.16
8	63.00	0.09	63.1	63.1	1.85	62.05	63.9	36	0.18
13	62.48	0.09	62.5	62.5	2	61.4	63.4	36	0.17
15	62.36	0.09	62.5	62.6	2.2	60.9	63.1	36	0.18
10	62.68	0.09	62.8	62.8	2.4	61.5	63.9	36	0.17
9	63.40	0.11	63.6	63.8	2.4	61.95	64.35	36	0.22
11	60.28	0.10	60.3	60.9	2.5	58.95	61.45	36	0.20
4	63.34	0.10	63.4	63.9	2.6	61.95	64.55	36	0.21
12	64.36	0.11	64.5	64.5	2.6	62.7	65.3	36	0.23
16	62.77	0.10	62.9	62.6	2.65	61.25	63.9	36	0.21
2	62.81	0.12	62.9	63.0	2.75	61.25	64	36	0.23
5	62.60	0.12	62.6	62.2	2.8	60.9	63.7	36	0.25
3	59.19	0.13	59.3	58.3	2.85	57.5	60.35	36	0.26
14	64.65	0.11	64.7	65.0	2.95	62.85	65.8	35	0.23
7	63.66	0.12	63.6	63.7	2.95	61.9	64.85	36	0.25
1	63.81	0.13	63.9	64.0	3.0	62.0	65.0	36	0.26

Table A4. Statistical Evaluation of Permeation Specimen Thickness Measurements: 80 mil Set, Welded

80 mil Range Sort	Mean (mil)	Std Error (mil)	Median (mil)	Mode (mil)	Range (mil)	Range (%)	Minimum (mil)	Min/Mean (%)	Maximum (mil)	Count	95% Confidence Level (%)	95% Confidence Interval (mil)
12	79.41	0.08	79.50	79.75	1.7	2.1	78.6	1.02	80.3	36	0.17	0.217
3	78.59	0.08	78.75	78.95	1.9	2.4	77.35	1.58	79.25	36	0.17	0.214
13	82.95	0.09	83.10	83.30	1.95	2.4	82.05	1.08	84	36	0.18	0.216
11	78.07	0.10	78.15	78.15	2.25	2.9	76.7	1.75	78.95	36	0.20	0.252
17	83.63	0.13	83.98	84.05	2.7	3.2	81.8	2.19	84.5	36	0.26	0.308
7	77.72	0.12	77.65	77.65	2.9	3.7	76.25	1.89	79.15	36	0.23	0.302
10	81.08	0.11	81.23	81.60	3.05	3.8	79.35	2.13	82.4	36	0.22	0.275
16	83.26	0.15	83.60	81.40	3.05	3.7	81.4	2.24	84.45	36	0.30	0.361
2	81.03	0.15	81.33	81.65	3.1	3.8	79.1	2.38	82.2	36	0.30	0.375
14	83.29	0.15	83.53	83.80	3.1	3.7	81.65	1.97	84.75	36	0.30	0.364
15	78.94	0.11	78.90	78.80	3.1	3.9	77.15	2.27	80.25	36	0.23	0.288
9	84.19	0.14	84.40	85.15	3.1	3.7	82.2	2.37	85.3	36	0.29	0.341
4	83.38	0.13	83.53	83.25	3.15	3.8	81.3	2.50	84.45	36	0.26	0.312
8	84.74	0.18	85.18	85.65	3.5	4.1	82.4	2.76	85.9	36	0.36	0.423
1	82.64	0.17	83.08	83.25	3.7	4.5	80.4	2.71	84.1	36	0.35	0.423
6	82.51	0.14	82.60	82.50	3.85	4.7	80.2	2.80	84.05	36	0.29	0.348
5	84.65	0.22	85.08	85.80	5.1	6.0	81.05	4.25	86.15	36	0.45	0.532

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